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TECHNICAL RESEARCH REPORT

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AIR MATERIEL COMMAND
UNITED STATES AIR FORCE
WRIGHT-PATTERSON AIR FORCE BASE, OHIO

PREPARATION OF BORON TRICHLORIDE
PART II PREPILOT AND PILOT PLANT PREPARATION OF FEED MATERIALS

OLIN MATHIESON PILOT PLANTS

OLIN MATHIESON CHEMICAL CORPORATION
ENERGY DIVISION
NIAGARA FALLS, NEW YORK

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PREPARATION OF BORON TRICHLORIDE PART II PREPILOT AND PILOT PLANT PREPARATION OF FEED MATERIALS

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I. SUMMARY

Prepilot and pilot plant feed preparation operations were directed toward the development of a suitable boric oxide-carbon feed material capable of chlorination to boron trichloride in a vertical-shaft fixed-bed or moving-bed reactor. The experimental program also included the development of carbon compacted feed for use in a secondary reactor. Mixtures of boron and carbon carrier together with a binder were first compacted into granules, almond shaped briquets, and tablets. Next the various feed formulations were sintered at temperatures ranging from 200 to 1300°F for periods of time ranging from 20 to 180 minutes in order to establish (a) optimum sintering conditions and formulations with respect to amount and type of carbon and boron carrier, and binder, and (b) sintering losses. Carbon sources (Witco furnace black, petroleum coke powder, and Gilsonite) and binders consisting of petroleum pitch sugar-water, unrefined sugar-water, starch-water and molasses water were evaluated. Boron carriers investigated were boric oxide, boric acid and sodium tetraborate. Pelletized feed was sintered with the use of batch and continuous equipment. Tests were also conducted to evaluate prototypes of commercially available equipment for compacting, drying, and sintering feed materials.

In general, laboratory tests showed that the most suitable boron carrier, carbon carrier, and binder were, respectively: 60 or 100 mesh technical boric acid powder, Witco Chemical Co. F-1 powdered and SRF beaded furnace black, and sugar-water. These tests also showed that mixtures of boric acid, furnace black and binder could be compacted into tablets, briquets, or granules to produce a hard easily handled material which upon sintering at 800 to 900°F yielded a hard porous solid granule containing 0.2 to 0.3 per cent hydrogen. Chlorination experiments showed that the sintered granules were highly reactive. Acceptable sintered carbon briquets and tablets were prepared by sintering mixtures of Witco carbon and sugar-water at 750°F for 1 hour. Hygroscopicity tests on sintered briquets showed that sintered feed is very hygroscopic, thus requiring appropriate storage facilities.

II. INTRODUCTION

In October 1955 process development operations were inaugurated for the purpose of developing a suitable boric oxide-carbon or sodium tetraborate-carbon feed capable of being chlorinated in a vertical shaft fixed- or moving-bed reactor to produce boron trichloride. Experimentation was conducted in prepilot and pilot plant size equipment. Overall operations consisted of; (a) preparing a mix consisting of the boron carrier (boric oxide, or boric acid, or sodium tetraborate) and carbon ("Witco" furnace black, petroleum coke or Gilsonite); (b) adding a binder, (water, sugar, sugar-water, starch water or pitch); (c) mixing these constituents in a ribbon or sigma mixer; (d) forming spherical feed by rolling in an inclined rotating drum or pressing the mixed feed into tablets or almond shaped briquets in appropriate machinery; and (e) drying or sintering the green (unsintered) compacted feed to decompose the binder and convert the boric acid to boric oxide or to convert the hydrated sodium tetraborate to the anhydrous form. Feed preparation studies are divided into two primary categories: (a) preparation of unsintered compacted feed (pelletizing) and (b) drying and sintering.

With specific boron and carbon raw materials and a specific carbon to boron ratio, small experimental batches of solids were mixed with varying amounts of binder to develop formulations capable of yielding acceptable green compacted feed. Experimental formulations producing acceptable unsintered compacted feed were sintered at elevated temperatures. Optimum formulations were scaled up to determine the effect of such variables as sintering time and temperature on sintering losses, residual hydrogen content, and mechanical characteristics. The program also included development of a carbon compacted feed for use in a secondary carbon-bed reactor*. In addition to the basic program described above, this investigation included oxidation of green compacted feed, hygroscopicity of sintered boric oxide-carbon feed, use of a rotary calciner to sinter green compacted feed, effect of predrying prior to sintering, and several alternate methods of producing feed with standard industrial equipment.

Criteria used to determine the acceptability of pelletized-sintered feed were:

1. Capability of the formulation used to produce sound unsintered pellets.

2. Resistance to disintegration during transfer from compacting process equipment to the sintering chamber.

3. Maintenance of shape during sintering; resistance to flow and fusing.

4. Resistance to deformation under a pressure of 1.5 p. s. i. (equivalent to 5 feet of bed in the chlorinator).

5. Resistance to disintegration when charged to the chlorinator.

6. Minimum of hydrogen content of sintered feed.

In order to designate the various solid feed formulations employed in the test program a code was developed. An explanation of this code is as follows:

Designation:	A	-	100	W	20	SuWa	(80 Wa)
	↓		↓ ↓	↓	↓	↓	↓
Item:	1		2	3	4	5	6

Item 1: Source of boron
A = boric acid, H_3BO_3
O = boric oxide, B_2O_3
NBO = sodium tetraborate, $Na_2B_4O_7$ (anhydrous)

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*Prepilot chlorination studies are summarized in the following reports Olin Mathieson Chemical Corp. Technical Reports OMCC-HEF-100 (1) and OMCC-HEF-158 (2)

Item 2: Per cent excess carbon over the stoichiometric quantity based on the reaction:

$$B_2O_3 + 3\,C + 3\,Cl_2 \longrightarrow 2\,BCl_3 + 3\,CO \qquad (I)$$

Example: A-100W etc. contains 123.6 g. boric acid (2 moles) which is equivalent to 1 mole of boric oxide plus 72 g. Witco carbon black, (6 moles)

Item 3: Source of carbon
W = Witco Chemical Company furnace black: F-1 powder for tabletted feed and SRF beaded black for briquetted feed
CG = calcined Gilsonite
PC = petroleum coke

Item 4: Weight per cent of the binder on a wet (acid or oxide and carbon and binder) basis

Item 5: Type of binder
K-150: Koppers Pitch, m. p = 150°F
K-200: Koppers Pitch, m. p. = 200°F
SuWa: sugar-water
BSuWa: brown sugar-water
SWa: starch-water
MWa: industrial molasses-water

Item 6: Weight per cent of water in the binder

A list of boron and carbon raw materials is presented in Appendix A; Figure 1 shows the boron to carbon ratio versus excess carbon in the feed formulation code.

III. PREPARATION OF FEED FORMULATIONS

A. Tabletting (Bench Scale)

1. Equipment and Procedure

Carbon and boron (boric oxide, boric acid or sodium tetraborate) tablets were prepared in accordance with the following operating procedures:

1. The carbon and boron carriers (raw materials) were premixed in a rotating drum.

2. The premixed dry ingredients were transferred to a sigma blade mixer.

3. The binder was prepared and slowly added to the premixed dry ingredients contained in the sigma mixer.

a. Pitch binder was prepared by chilling K-150 or K-200 pitch with Dry Ice and then pulverizing and screening the chilled pitch.

b. Starch-water, molasses-water, or sugar water was mixed to obtain a homogenous solution before addition to the dry ingredients.

4. The "wet" feed was mixed for approximately 1 hour.

5. "Wet" feed was removed from the mixer and allowed to dry at ambient temperature if necessary.

6. The dry mix was screened through a 14 mesh standard screen.

7. The mix was pressed into 1/4 to 3/8 inch diameter by 1/4 inch long tablets with a Stokes model (A3) Tabletting Machine.

8. The "green" tablets were sintered in a Cenco-Cooley bench size electric muffle furnace. Process flowsheets for the preparation of granules, tablets, and briquets are presented in Figure 2.

Experimental formulations prepared to determine optimum binder content with respect to green tablet strength characteristics were homogenized with a mortar and pestle instead of the sigma mixer because each batch consisted of only 250 grams.

Results obtained from the tablet development studies for various sources of boron, carbon, and binders are summarized in Table I. Table I also includes sintering characteristics. Sintering characteristics may not be divorced from feed compacting with respect to tablet development studies. A number of feed formulations yielded acceptable "green" (unsintered) tablets which deformed and/or fused upon sintering.

2. Preparation of Boric Oxide-Carbon Tablets with K-150 or K-200 Pitch

Pellet development experiments were initiated with the use of 60 mesh Pacific Coast boric oxide, Witco F-1 furnace black, and Koppers pitch having a melting point of either 140 - 150°F (K-150) or 200 - 220°F (K-200). Optimum formulations containing various amounts excess carbon were developed. Data obtained from these studies are summarized in Table II. Two criteria were used to evaluate the tablets. The first consisted of a drop test and is a modified American Society for Testing Materials test for the determination of the relative strength of tablets before sintering. The test procedure consisted of dropping a number of tablets from various heights and noting the maximum height from which 75 per cent of the tablets maintained their shape. The other criterion used consisted of heating the green tablets to 1110°F (600°C) and noting the physical characteristics of the sintered tablets. Acceptable tablets should neither deform nor fuse during the sintering operation. Figure 3 relates the effect of excess carbon on results of the drop test and sintering test for formulations containing boric oxide and K-150 pitch. It is noted that the maximum tablet strength before sintering and the most satisfactory pellets after sintering did not coincide at a common percentage of excess carbon. When K-200 was used as the binder material (Figure 4) very little change in the before-sintering strength occurred until approximately 200 per cent excess carbon was used, after which a rapid decrease occurred. Figure 4 also shows that the optimum

Witco F-1 carbon content is approximately 165 per cent and that this is nearly the same as for formulations with K-150 pitch as binder. Although the formulations 0-150W-25K150, 0-100W-25K150, 0-150W-25K200 yielded acceptable tablets from a standpoint of physical characteristics both before and after sintering, cost estimates indicated that savings could be realized by using boric acid rather than boric oxide as the source of boron.

Results obtained from tablet preparation studies with boric acid, Witco F-1 carbon or Great Lakes Carbon Co. petroleum coke, and K-150 or K-200 pitch binder are summarized in Table III. Optimum formulations developed from this phase of study were included in Table I. Figure 5 shows the effect of various amounts of K-150 pitch on the strength of tablets formed from boric acid and 10 per cent excess Witco carbon. This figure shows that the formulation resulting in the lowest number of tablets broken during preparation also produce the firmest sintered tablets. K-150-bound formulations prepared with boric acid and containing as much as 100 per cent excess carbon, exhibited excessive fusing during sintering. For this reason, and because of excessively high sintering temperatures required to eliminate sulfur in the sintered tablets (originating from the pitch), the use of starch-water and sugar water as the binder was investigated.

3. Preparation of Boric Oxide-Carbon Tablets from Boric Acid and Starch-Water Binder

Boric oxide-Witco carbon tablets with starch-water solution as the binder were prepared with excess carbon contents ranging from 100 to 1000 per cent. Results compiled in Table I show that some fusing was observed for sintering temperatures as low as 750°F. Also physical characteristics were hard to duplicate from batch to batch. However, the substitution of starch-water for K-150 resulted in considerably less fusing.

4. Preparation of Boric Oxide-Carbon Tablets from Boric Acid and Sugar-Water Binder

The utilization of sugar water as the binder in the preparation of boric oxide-Witco carbon tablets represented a substantial improvement from the standpoint of green and sintered tablet characteristics. Data presented in Table I show that acceptable unsintered tablets were produced with from 50 to 1000 per cent excess carbon. With the exception of the 50 per cent excess carbon formulation (A·50W15SuWa (80 Wa)) which fused on sintering at temperatures as low as 1110°F, all these formulations produced hard and undeformed sintered stock which did not fuse in the sintering tray at temperatures at least as high as 1290°F. Optimum sugar-water binder contents are illustrated in Figure 6.

5. Use of Alternate Sources of Sugar

As part of the overall program to determine the cheapest raw materials for the chlorination process tablet development studies were conducted with brown sugar and molasses instead of the refined form of sugar. Brown sugar-water solutions ranging in concentration from 20 to 40 per cent sugar and total binder contents ranging from 5 to 25 per cent

(on a wet basis) were tested utilizing boric acid and Witco F-1 as the source of boron and carbon, respectively. Excellent unsintered tablets for the formulation A·100W·20B SuWa (60Wa) were obtained which did not exhibit any tendency to fuse or deform when exposed to temperatures as high as 1110°F for 60 minutes. However, the equivalent formulation with refined sugar, A·100W·20 SuWa (80Wa) required only half as much sugar; thus the use of brown sugar would not be economically feasible. In addition, subsequent sintering tests showed that higher sintering temperatures were required to reduce the residual hydrogen content to 0.24 per cent.

A satisfactory A·100W tablet was developed with industrial molasses-water as the binding agent. Tabletted feed of the formulation A·100W·20MWa (60Wa) exhibited excellent sintering characteristics comparable to A,100W formulations in which starch-water and sugar-water were the binders. However, a significant disadvantage of industrial molasses is its high ash content: approximately 8.4 per cent. Sintered A·100W·20MWa (60Wa) tablets contained 2.08 to 2.70 per cent ash which is believed to be sufficient to adversely affect reactivity or fusion characteristics. For these reasons the utilization of molasses as a binding agent is not advisable.

6. Elimination of Sugar in the Binder

It was possible that sugar could be eliminated from the sugar-water binder since boric oxide exhibits binding characteristics. Accordingly an A·100W experimental mix was developed with the use of only water. Although these tablets showed excellent physical characteristics after a sintering treatment at 1000°F they exhibited very poor strength characteristics in the unsintered condition. This experiment indicates that water alone should not be used as the binder.

7. Use of Alternate Sources of Carbon

As part of the general program for investigating alternate carbon carriers with emphasis on cost reduction, formulations containing boric acid and sugar-water were prepared utilizing petroleum coke, Witco SRF carbon beads, and Gilsonite (a form of petroleum coke). Acceptable unsintered tablets containing 100 and 200 per cent excess petroleum coke were developed. Upon sintering at 1000°F severe fusion was observed which was attributed to the inability of the hard dense carbon particles to absorb and disperse the boric oxide. The use of petroleum coke as the carbon source is not recommended.

In view of anticipated use of Witco SRF carbon beads in pilot plant operations for preparation of briquetted reactor feed, formulations containing 50 and 100 per cent excess SRF Witco carbon were prepared and tabletted. Witco SRF furnace black (1/16 inch diameter beads) are prepared from Witco F-1 powder by a balling process. In each case the ultimate particle size is 70 millimicrons. Results from these experiments, which are presented in Table I, show that excessive fusing occurred for both optimum formulations at a sintering temperature of 1000°F. This behavior suggested that the Witco SRF beaded carbon, having a much higher apparent particle size, was unable to absorb or disperse the boric acid

powder during mixing. Furthermore, it was indicated that adequate dispersion of boric acid in carbon on a micro scale is required to minimize fusing. Excessive fusing of A·100W SRF25K150 tablets was observed earlier in the development program when inadequate inventory of F-1 powder necessitated utilization of SRF Witco carbon.

Calcined Gilsonite coke (American Gilsonite Company) which is obtained as a residue from shale was investigated because of its lower cost. All formulations containing 100 per cent excess carbon (A·100CG) and sugar-water binder were too wet and thus incapable of being formed into tablets. Elimination of water in the binder yielded a mix capable of being pressed into tablets. However, A·100CG tablets prepared with only sugar as the binding medium were rather weak before sintering and fused severely when sintered at 1000°F. Again, the fusing may be attributed to the inability of the Gilsonite particles (100 mesh) to disperse the boric acid. Calcined Gilsonite is not recommended for use as carbon source for feed preparation.

8. Preparation of Anhydrous Sodium Tetraborate-Witco Carbon Tablets with Sugar-Water Binder

Tablet development studies were expanded to include feed prepared with sodium tetraborate as the boron carrier. Initial studies were conducted with Razorite, ($Na_2B_4O_7 \cdot 4H_2O$). The optimum 100 per cent excess carbon formulation, NBO·100W·45SuWa (80Wa), produced acceptable compacted feed which fused severely upon sintering at 1000°F. Formulations containing 200 and 300 per cent excess carbon also fused severely. Development studies with granular Razorite were abandoned and instead anhydrous borax was used as the boron carrier. Addition of water to anhydrous borax-carbon mixtures resulted in the formation of hydrates of borax which caused the mix to set similar to cement. Small batches of borax-carbon tablets containing between zero and 200 per cent excess carbon were successfully tabletted by minimizing the water content of the binder. Whereas the formulations containing between zero and 50 per cent excess carbon yielded fairly good sintered stock, mixes containing between 100 and 200 per cent excess carbon almost disintegrated when sintered at 1000°F. Failure to develop a satisfactory anhydrous borax-carbon tablet containing 100 to 200 per cent excess carbon was attributed to increased water requirements and partial loss of natural binding action of sodium tetraborate at the higher carbon contents. The use of K-150 coke pitch instead of sugar-water solutions was found satisfactory from the standpoint of pelleting; when sintered, oil- or pitch-bound formulations fused.

9. Preparation of Carbon Tablets from Witco F-1 Carbon with K-150, Pitch, Sugar-Water or Starch-Water Binders

The data contained in Table I show that attempts to develop an acceptable carbon-base tablet with Witco F-1 powder and K-150 pitch were unsuccessful. Witco-K-150 pitch formulations became very fluffy during mixing which resulted in a thin, undersized, and weak tablet.

The utilization of either sugar or starch solutions as the binder yielded satisfactory green and sintered feed. The optimum formulation 67W 33SuWa (70Wa) proved to be slightly superior to that of 67W 33SWa (70Wa) in that before sintering strength was somewhat better. In spite of the development of acceptable sugar-water and starch-water bound carbon tablets the optimum formulations were weaker than boric oxide-carbon formulations. Dusting and cracking occurred during sintering which would necessitate recycling a substantial amount of sintered stock. The reduction in strength after sintering was attributed to the absence of boric oxide which is a natural binder.

B. Briquetting (Pilot Plant)

1. Equipment and Procedure

One hundred pound batches of almond shaped boric oxide-carbon and carbon briquets were prepared in accordance with the following procedure:

1. Sixty-mesh boric acid was pulverized in a "Mikro-Pulverizer" size No. 1 SH, manufactured by the Pulverizing Machinery Company.

2. A predetermined amount of Witco SRF beaded carbon and Witco pulverized boric acid was charged to a ribbon mixer (type B. J. H. Day Co.).

3. The sugar-water or starch-water binder solution was added slowly to the mixing dry chemical.

4. Mixing was continued for 30 minutes or until the constituents were thoroughly mixed (as determined by visual observation). The mix was adjusted to proper consistency by adding small amounts of water or carbon.

5. The blended mix was removed from the ribbon mixer and charged to the briquettor feed hopper.

6. The mixed feed was pressed into 1 by 3/4 by 1/2 inch briquets with a Komarek-Greaves model 4192 briquetting machine having a capacity of from 51,840 to 129,500 briquets per hour. (Fine, particles or undersized or imperfect briquets are separated from acceptable stock by allowing the briquets dropping from the briquettor to pass over an inclined screen).

7. The briquets were then charged to an electrically-heated, 10-tray (capacity, 25 lb./tray) Special Gehnrich Bench type oven manufactured by the W. S. Rockwell Company.

8. The briquets were sintered at the following temperatures under a continuous nitrogen purge:

400°F for 1/2 hour
500°F for 1/2 hour
600°F for 1/2 hour
900°F for 1-1/2 hour

9. The sintered briquets were removed from the oven, screened to remove particles and defective stock and stored in sealed 10 gallon cans.

Several batches of unsintered briquets were transferred to the prepilot-group for sintering and reaction evaluation tests. Also, samples from several batches were heated in a laboratory type muffle furnace to evaluate fusing characteristics.

2. Preparation of Boric Oxide-Carbon Briquets with Starch-Water Binder

Briquet development operations were conducted to develop a starch-water bound boric oxide-carbon briquet containing 100 per cent excess carbon from 60-mesh technical grade boric acid powder (Pacific Coast Borax) and SRF-Witco beaded carbon. Results from these experiments are presented in Table IV.

Binder ratios of from 0.1 to 0.35 starch/boric acid + carbon were investigated with the use of uncooked starch. Overall formulations tested ranged from A·100W·25.5 SWa (88Wa), S/BA+C = 0.04, Wa/BA+C = 0.30 to A·100W·45.1 SWa (57.3 Wa), S/BA+C = 0.35, Wa/BA+C = 0.47. Increasing the S/BA+C ratio from 0.1 to 0.35 was accompanied by a decreased in the strength of unsintered briquets. Good unsintered briquets were obtained with the following formulation: A·100W·25.5SWa (88Wa), S/BA+C = 0.04, Wa/BA+C = 0.30. However, an A·100 starch-water bound briquet having as good strength unsintered as sugar-water bound formulations could not be attained. Upon sintering at 1000°F, starch-water bound formulations retained their original shape and showed no evidence of fusing or deforming. However, when exposed to 1300°F under a weight equivalent to 4 feet of bed, deformation and fusing was observed.

Several A·100W experimental batches were prepared with cooked starch-water solutions as the binder. Formulations investigated ranged from A·100W·24.4SWa (87.5Wa), C/BA+C = 0.04, Wa/BA+C = 0.28 to A·100W·48.8SWa (89.5 Wa), S/BA+C = 0.1, Wa/BA+C = 0.85. Cooked starch-water formulations having high Wa/BA+C ratios were too damp and could not be briquetted. Except for formulations having low starch and water ratios, excessive balling and granulation occurred during mixing in the Day mixer. The formulation A·100W·24.8SWa (87.5Wa) yielded unsintered briquets superior to those using uncooked starch but inferior to those sugar water binder solutions. Sintering and muffling (at 1300°F) characteristics for cooked starch-water formulations were identical to those with uncooked starch-water as binder.

3. Preparation of Boric Oxide-Carbon Briquets with Sugar-Water Binder

Initially, experimentation was confined to the development of a boric acid Witco SRF carbon-sugar water briquet containing 100 per cent excess Witco carbon. Formulations investigated were in the range A·100W·17SuWa (80Wa), Wa/BA+C = 0.164 and SU/BA+C = 0.041, to A·100W·31.1 SuWa (33.3Wa), Wa/BA+C = 0.152 and Su/BA+C = 0.297.

Batches having a low binder content such as A·100W·17SuWa (80Wa) exhibited poor strength before sintering. However, the formulation A·100W·25 SuWa (85Wa) produced excellent unsintered briquets which exhibited no or very little fusing when sintered at 1000°F. Yet briquets of the last two formulations deformed and fused when muffled at 1300°F under a weight equivalent to 4 feet of briquets. Increasing the total binder content and the sugar/boric acid + carbon ratio yielded excellent unsintered briquets which sintered well and did not deform or fuse during the muffling test. This optimum formulation was A·100W·31.1 SuWa (33Wa), Wa/ BA+C = 0.15 and Su/BA+C = 0.30. Increasing the total binder content to 34 per cent, A·100W·34SuWa (61.4 Wa) gave a mix that was too wet.

In preparing A·100W briquets with sugar-water binder, sintering characteristics were hard to duplicate. That is, some batches exhibited a tendency to fuse during the muffle test and, at times, during sintering. Whenever a new batch of raw materials was utilized, a slight adjustment in the binder content was necessary to produce a suitable mix. Although quantitative data were never obtained, particle size and strength characteristics of Witco SRF beaded carbon seems to have a significant influence on binder requirements and sintering characteristics.

It was theorized that the sintering characteristics were functions of the boron density and carbon content of the sintered briquets. Accordingly a series of 20-lb. mixes varying from 6 per cent excess carbon (A·6W·20SuWa (60Wa)) to 224 per cent excess carbon (A·224W·32.8 SuWa (41.2 Wa)) were briquetted and sintered. Results from these tests are shown in Figure 7 which compares the sintering characteristics as a function of carbon content and boron density of sintered briquets. Data summarized in Table V under "Variation of Per Cent Excess Carbon" are based on analytical results of sintered briquets. Figure 7 shows that above 150 per cent excess carbon no fusing is encountered. The formulation A·150W·25SuWa (70Wa), Su/BA+C = 0.10 and Wa/BA+C = 0.234 yielded excellent sintered briquets and this formulation was used to produce briquets for chlorination in the pilot plant reactor Although this formulation required adjustment to compensate for inconsistancies encountered in different batches of raw materials, briquets produced were sound and fusing was not observed.

4. Preparation of Carbon Briquets with Starch-Water Binder

Formulations investigated for carbon briquet development studies with starch-water binders ranged from 65W·35 SWa (81.5 Wa) S/C = 0.1 and Wa/C = 0.44, to 47.25W·52.75SWa (68.8 Wa), S/C = 0.35 and Wa/C = 0.77. (Table VI) Almost all briquets of the latter formulation fractured or disintegrated during sintering, presumably because of excessive binder. Briquets of the former formulation exhibited a considerable improvement in sintering characteristics; however, after heating in the muffle furnace to 1300°F, they were crushed very easily. No such difficulty was encountered during carbon-sugar water briquet development. The use of starch-water solutions as binding media is not recommended.

5. Preparation of Carbon Briquets with Sugar-Water Binder

Results for SRF Witco carbon-sugar water briquet development studies are presented in Table VII. Sugar/carbon and Water/carbon ratios were varied. The formulation 69W·31SuWa (66.7Wa) produced unsintered briquets having very poor mechanical characteristics. This formulation is almost identical with the optimum formulation for tabletized feed 67W·33SuWa (70Wa) for which F-1 Witco powder was used. Increasing the sugar/carbon and water/carbon ratios to 0.35 and 0.25, respectively, considerably improved the strength of unsintered and sintered briquets. The optimum formulation 59W·41SuWa (64Wa) was used to prepare carbon briquets for the chlorination pilot plant. The significant increase in binder requirements may be attributed to greater particle size (SRF vs F-1) and the difference in mechanical equipment used to compact the feed. Regardless of the formulation, carbon-sugar water briquets tended to break when muffled at 1300°F.

Mixes 84A and 84B (see Table VII) were prepared to determine the effect on binder requirements of premixing the Witco SRF beads in the ribbon mixer. In experiment 84B the SRF carbon was premixed for approximately 1/2 hour before adding the binder. The same amount of sugar-water solution was required for both 84B and the control experiment 84A (no premixing).

C. Rotary Granulation

Preliminary feasibility studies indicated that the use of spherical feed for the chlorination reactor would result in a lower cost of feed preparation. Therefore, feed preparation studies were expanded to include the development of spherical granular feed material. Although some effort was expended to develop satisfactory borax-Witco carbon and Witco carbon granules, approximately 95 per cent of rotary granulation experiments were confined to boric acid-Witco carbon-sugar water-formulations.

1. Equipment and Operating Procedures

Three different rotary granulators were used in the program. Preliminary feasibility experiments were conducted with a 4-quart inclined Abbe Jar Mill rotating at approximately 60 r.p.m. Water was introduced into the jar mill containing the bed of dry materials through either a pneumatic nozzle or a pipette. Results with the jar mill (designated as RG-1) were encouraging and the process was scaled up to a 12-inch diameter by 16-inch long rotary granulator to accomodate 5 pound batches of dry mix. Figure 8, shows the primary components of this installation. A production unit, Rotary Granulator RG-111 (Figure 9) 36 inches in diameter by 14 inches long was fabricated having a capacity of 40 to 50 pounds of granules, in order to prepare granular feed for the pilot plant reactor. In order to produce balls of more constant diameter and to allow pre-drying of the granules with a blast of hot air, a 55 gallon drum for the purpose was installed at the discharge end of RG-111.

Operating procedure for the Abbe Jar Mill, RG-1, was as follows:

1. Two-hundred thiry grams of premixed dry solids (boric acid, Witco F-1 carbon and sugar) were charged into the jar mill.

2. A predetermined amount of water was slowly added to the dry mix contained in the rotating jar.

3. The mill was operated so as to produce balls of proper diameter.

4. The wet granules were sampled for moisture determination.

5. The remaining wet granules were dried at 194°F for 1 hour and sintered at 1000°F for an additional hour.

6. The sintered granules were screened; screened fractions were submitted for boron, carbon and hydrogen analysis.

Operating procedure for the 12 inch diameter by 6 inches long rotary granulator, RG-II, was as follows:

1. Five pounds of dry mix was charged to the rotary granulator.

2. Sufficient water was slowly added to yield granules of desired size.

3. The mix was allowed to granulate after addition of water so as to produce granules of satisfactory size.

4. The wet granules were sampled for determination of moisture.

5. The wet granules were dried at 194°F for 1 hour and screened to determine particle size distribution.

6. The screened granules were sintered at 1000°F for 1 hour.

7. The sintered granules were separated into the following fractions: 6 mesh to 1/4 in.; 1/4 to 1/2 in.; 1/2 to 3/4 in.; 3/4 to 1 in.

8. The sintered fractions were sampled for determination of boron, carbon and hydrogen content and stored in sealed containers.

The operating procedure for the RG-III unit, 36 inches in diameter by 14 inches long, was as follows:

1. Approximately 25 pounds of dry mix, screened through a 14 mesh sieve was charged into RG-III and premixed for 15 minutes.

2. Water was added to the rotating dry mix by means of a flat, spray nozzle.

3. The mix was allowed to granulate after the water addition; the sides of the can were knocked with a rubber mallet.

4. The moisture content of the wet granules was determined.

5. Wet granules were dried at 194°F for 1 hour.

6. Dried granules were screened into 1/4 in., 1/4 to 1/2 in., and 1/2 to 1 in. fractions.

7. The individual fractions were sintered at 1000°F for 1 hour.

8. The individual sintered fractions were sampled for boron, carbon and hydrogen determination and stored in sealed containers.

Various modifications of the operating procedures were instituted to determine their effect on granulation characteristics.

2. Preparation of Boric Oxide-Witco Carbon Granules with Sugar-Water Binder

Preliminary evaluation experiments showed that the water spray pattern was very important. If the water binder was sprayed on to the walls, excessive buildup of solids occurred. This difficulty was eliminated by making sure that the water was directed into the bed of dry feed and by rapping the rotating granulation drum with a rubber mallet. Initial granulation experiments with the RG-III equipment (which incorporated six flights and no scraper blade) were accompanied by excessive buildup on the chamber walls. Also the granules were inferior to those produced with RG-II equipment with respect to sphericity and surface characteristics. Increasing the batch weight to 33 pounds (dry) resulted in acceptable feed.

a. Effect of Sugar Content on Granulation Characteristics

Granulation experiments G-12 to G-17 were performed to show the effect of varying sugar binder content on granulation characteristics of A-100W mixes. Results from these experiments are summarized in Table VIII. Granulation apparatus RG-I was used for this series of tests. Water was added from a pipet to the boric acid-Witco carbon-sugar dry mix contained in the rotating Abbe Jar Mill. The following data from Table VIII were abstracted to show the effect of sugar content on granule size:

Per cent sugar	2	4.8	10
Per cent of granules 1/4 in. and larger	5	36	85

These data show that as the sugar content was increased from 2 to 10 per cent, an appreciable increase in particle size was effected. Data presented in Table VIII show that increasing the sugar content also resulted in an increase in the residual hydrogen content of the sintered granules. Granulation experiments G-19 to G-51, results of which are shown in Table IX, were conducted for the following purposes: G-19 to G-21, effect of sugar content on water requirement; G-22 to G-27, effect of formulation on sugar requirement; G-27 to G-32, granulation directly in a sigma mixer; and G-37 to G-51, production experiments for A·100W granules, 1/8 to 1/4 in. and 1/4 to 1/2 in. size fractions for use as prepilot scale chlorination reactor feed stock. Comparing the water added in experiments G-20, G-20RR and G-21 (no sugar in dry mix) with experiments G-12 to 17, it appears that decreasing the sugar content of the dry mix raises the water requirement. The formulation A·100W containing about 9 per cent sugar requires approximately 33 per cent water whereas the same formulation without sugar needed 40 per cent water on a dry mix basis. However, the following table suggests that the total binder content for A·100W formulations is not dependent on the relative amount of sugar contained in the binder:

Per cent sugar, dry basis	0	2	4.8	10
Per cent sugar, wet basis	0	1.5	3.6	7.7
Per cent total binder, wet basis	29	24.5	28.6	30.6

b. Effect of Granule Diameter on Boron, Carbon and Hydrogen Content

Analytical results for boron, carbon, and hydrogen contents of several size fractions are shown in Table X. Residual hydrogen data from earlier experiments (G-12 to 17, jar mill) were presented in Table IX. These data show that the small size particles contain less boron and more carbon than the larger fractions. This phenomenon is prevalent because carbon is more difficult to agglomerate. The data for residual hydrogen as a function of ball size suggests, at first approximation, that decreasing the particle results in a slight increase in the residual hydrogen content. One would normally expect the smaller size granules to contain less hydrogen because of the increased ease of hydrogen removal with decreased particle size. The higher hydrogen content of smaller granules is attributed to absorption of moisture during preparation of the sample. Improvements in sampling techniques showed that the residual hydrogen content of sintered granules was reduced to 0.27 per cent after sintering for 1 hour at 1000°F.

c. Effect of Carbon Content on Binder Requirement

The data contained in Tables IX and X show that the binder content, for various levels of per cent excess carbon, was as follows:

B/C wt. ratio	0.40	0.30	0
Formulation	A·50W·24.7 SuWa(85Wa)	A·100 W. 28.6 Wa	56.3 W·43.7 SuWa(86Wa)
Per cent excess carbon	50	100	∞
Per cent total binder Requirement (wet basis)	24.7	28.6	43.7

These data are plotted in Figure 10.

d. Effect of Boric Acid Raw Material Particle Size on Granulation Characteristics

Several granulation experiments were conducted to determine the feasibility of using granular (20 mesh) boric acid instead of the more expensive 100 mesh impalpable grade. Tests disclosed that suitable yields of 1/4- to 1/2-inch diameter granules were not obtainable. The substitution of Pacific Coast Borax boric acid "powder" for the 100 mesh impalpable acid produced granules comparable to those of 100 mesh boric acid.

e. Use of Sigma and Ribbon Mixers to Improve the Homogeneity of Solids

Experiments G-27 to G-32 summarized in Tables IX and X represent attempts to granulate directly in a sigma blade blender. Moisture requirements were lower but additional rolling in a rotary drum was required to attain satisfactory agglomerates. However, a considerable improvement was noticed with respect to boron distribution in the various particle sizes as shown in Table X, which is the result of better dry solids mixing. Encouraging results from these laboratory experiments led to the initiation of slurry granulation tests which were performed by the Link Belt Co. Results from these outside tests are presented in Section III-E

f. Optimum Formulations and Operating Conditions

The following optimum formulations and operating conditions were established:

RG-II, 12 in. diameter by 16 in. long:

Formulation	A·100W granules
H_3BO_3	2.72 lb.
Witco F-1 powder	1.59 lb.
Water	1.83 lb.
Water rate	12 lb./hr.
Method of water addition	flat spray nozzle, 30 p.s.i.g.

RG-III, (36" diameter x 14" long):

Formulation	A·100W granules
H_3BO_3	20.8 lb.
Witco F-1 powder	12.2 lb.
Water	13.2 lb.
Water Rate	1.13 lb./min.
Method of Water addition	flat spray nozzle
Total Mixing Time	54 min.
Dry mixing time	30 min.
Time of water addition	11.7 min.
Wet mixing time	12 min.
Granulator Speed	15 r.p.m.
Granulator flights	6 1-1/2 inch angle iron

Particle distribution	70 per cent 1/4 to 1/2 inch diameter granules
Recycle Rate	30 per cent
Production rate, 1/4 to 1/2 inch diameter stock	
On Total mixing time basis	6.6 lb./ft.^{2}/hr.
On Water addition and wet mixing time*	15.2 lb./ft.^{2}/hr.

3. Preparation of Witco Carbon Granules with Sugar-Water Binder

Granulation experiments G-23 and G-23R were performed using only carbon and sugar as dry mix. Satisfactory-wet granules equivalent to the formulation 56.3W·43.7 SuWa (86Wa) were prepared. This formulation contains a considerable amount of binder with respect to the optimum tabletted feed formulation 67W·33 SuWa (70Wa). However, it is comparable to the optimum carbon briquet formulation 59W·41 SuWa (64Wa) as far as total binder content is concerned. Upon sintering, the Witco carbon base granules cracked and dusted rather severely. A satisfactory carbon granule was not obtained.

4. Preparation of Borax-Carbon Granules with Sugar-Water Binder

A limited number of rotary granulation experiments were conducted to investigate the granulation characteristics of anhydrous borax-Witco carbon mixtures. Rotary granulator RG-III was used. Initially, 33-pound batches of borax and carbon of the formulation NBO·100 W were found to require approximately 36 pounds of spray water equivalent to 109 per cent of the original dry mix or 40 per cent of the mix figured as sodium tetraborate decahydrate ($Na_2B_4O_7 \cdot 10H_2O$) and carbon. Considerable heat was evolved during the water addition phase. The best yield of 1/8 to 1/2 inch diameter nodules was 63 per cent and surface characteristics of the unsintered granules were comparable to those obtained from the boric oxide-Witco carbon experimentation. Attempts to sinter these granules at 1000°F resulted in formation of fused masses (chunks) and excessive carbon losses. The excessive fusion is related to the low melting points of hydrated borates. Similar difficulties were encountered during anhydrous borax-Witco carbon tablet development operations. Extensive granulation studies on anhydrous borax-carbon formulations were not conducted due to severe fusion during chlorination. A satisfactory anhydrous borax-Witco carbon granule was not developed.

D. Drying and Sintering

1. Boron, Carbon and Hydrogen Losses for Boric Acid-Carbon Tablets

a. Scope, Operating, and Analytical Procedure

In order to establish optimum operating conditions during sintering (drying), approximately 400 grams of tablets contained in a stainless steel tray were exposed to temperatures between 200°F and 1290°F for periods of time ranging from 20 to 180 minutes. To determine the effect

*The dry mixing could be accomplished more effectively in a separate mixer

of a nitrogen purge on boron and carbon losses and residual hydrogen content, several experiments were conducted with and without the purge. Equipment and operating procedure were described in Sections III A, B and C. Boron, carbon and hydrogen losses under various sintering conditions were established from analyses and weighings before and after sintering. Precautions, such as grinding the sample of sintered feed in a dry box and sealing the sample jar with tape, were taken to minimize absorption of moisture. Sulfur content and losses were determined for tablets in which K-150 or K-200 pitch was the binder. Ash content of formulations containing molasses-water binder solutions was also determined.

Conventional analytical methods were used to determine boron, carbon, hydrogen and sulfur content. Boron and Carbon were determined by the following procedure:

1. A weighed amount of pulverized feed was placed in a fritted glass crucible.

2. The pulverized sample was washed with acetone if it contained K-150 or K-200 pitch.

3. The sample was washed with hot acidified water to extract the soluble boron as boric acid.

4. The filtrate was titrated with standard sodium hydroxide with the use of mannitol method to determine soluble boron.

5. The residue contained in the fritted glass crucible was dried at 230°F and weighed. Per cent carbon was found by difference. The dried residue in the fritted glass crucible was assumed to be all carbon.

Hydrogen was determined by conventional macro or micro combustion methods. Sulfur was determined by the Parr bomb method; (fusion with sodium peroxide, colorimetric determination).

b. K-150 Pitch Bound Tablets

Losses for A-100W tablets prepared with use of K-150 pitch as binder at various sintering temperatures and times are summarized in Table XI and Figure 11. In spite of the high degree of scatter exhibited by the data in Figure 11, it appears that 1200°F is the approximate optimum temperature for the removal of sulfur and hydrogen from the tablets.

Water present in the solid feed tablets will react with boron trichloride formed during chlorination to liberate hydrochloric acid and boric oxide in accordance with the following reactions:

$$B_2O_3 + 3\ Cl_2 + 1\ 1/2C \longrightarrow 2BCl_3 + 3/2\ CO_2 \qquad (2)$$

$$CO_2 + C \rightleftharpoons 2CO \qquad (3)$$

$$2\ BCl_3 + 3H_2O \longrightarrow B_2O_3 + 6HCl \qquad (4)$$

The formation of hydrochloric acid and boric oxide is highly undesirable from three standpoints: (a) reduction in boron trichloride yield, (b) increase in corrosion due to presence of hydrochloric acid, and (c) deposition of white solids (primarily boric oxide) which tend to plug the effluent gas lines. The third undesirable characteristic resulted in the premature shutdown of numerous chlorination experiments. Residual hydrogen content versus sintering time curves presented in Figure 11 show that the hydrogen content for A•100W 20K-150 tablets sintered at 1200°F for 60 minutes was approximately 0.64 per cent. This hydrogen content was equivalent to 5.7 per cent water and would cause a ten per cent reduction in boron trichloride yields. In view of the relatively high residual hydrogen content, high sintering temperatures, the uncertainty regarding complete elimination of sulfur, and the tendency to fuse and deform during sintering, development studies of K-150 pitch as a binder for boric oxide-carbon tablets were discontinued.

c. Sugar-Water Bound Tablets

Sintering losses for boric oxide-Witco carbon tablets prepared with sugar-water solutions are shown in Table XII. Sintering loss data as a function of time for various sintering temperatures are illustrated in Figures 12 A through F. In general, results are quite erratic. For example in Figure 12B (batch 28D), the over-all sintering loss at 1200°F was higher than at 1290°F. However trends shown by the data indicate that the sintering treatment of 1 hour at approximately 930°F was necessary for consistent removal of hydrogen to 0.25 per cent. Under these conditions, boron and carbon losses were about 15 and 7 per cent respectively. Negative carbon losses (indicating carbon pickup) may be attributed to analytical procedures and the method of calculating carbon losses. Reference to the analytical procedure outlined in the previous section shows that the binder was eliminated from the unsintered briquets either by washing with carbon tetrachloride or acidified water. Consequently this carbon content did not include carbon from pitch, sugar, or starch binder.

Sintering tests conducted with use of a nitrogen purge passed through the sintering chamber, are summarized in Table XII, and sintering data are plotted in Figures 12 D, E and F. Although no consistent beneficial effect of the inert atmosphere was observed, it is felt that on a larger scale a nitrogen atmosphere would minimize carbon losses due to oxidation.

d. Brown Sugar-Water Bound Tablets

Sintering tests on the formulation A·100W·20BSuWa (60Wa) at 930, 1110, and 1290°F showed that a temperature of 1110°F was required for removal of hydrogen to 0.24 per cent. The increased sugar requirement (8 per cent for brown sugar, 4 per cent for refined sugar) and the higher sintering temperature would exceed the cost differential between refined and brown sugar. Therefore, the use of brown sugar-water solutions as the binder is not feasible.

e. Molasses-Water Bound Tablets

Sintering data for molasses-water bound tablets are shown in Table XIII and are reproduced graphically in Figure 13. Although sintering losses compare quite favorably with those for starch-water and

sugar-water bound tablets, the high residual ash contents, 2.08 to 2.70 per cent, shown in the footnote of Table XIII discourage serious consideration of molasses as a binder. A high ash content not only can adversely affect reactivity of the tablets but rapid buildup of ash in the residue will occur thus making the recycle of residue costly.

f. Starch-Water Bound Tablets

Sintering data for boric acid-carbon tablets prepared with an uncooked starch-water solution are presented in Tables XIV and Figures 14 A through D. These tests were conducted with and without a nitrogen purge at temperatures ranging from 300°F to 1290°F and for residence times of from 20 to 180 minutes. The data show that starch-water bound tablets sintered at 750°F for 1 hour had residual hydrogen contents of less than 0.25 per cent. Boron and carbon losses were essentially the same as those shown for sugar-water bound tablets. In spite of the slightly lower sintering temperature required for starch-water bound boric oxide-carbon tablets, production batches of tablets and briquets for chlorination were prepared with the use of sugar-water binder because of improved unsintered strength, and the absence of deformation at sintering temperatures.

g. Relative Sintering Losses of K-150 Pitch, Molasses-Water, Sugar-Water and Starch-Water Bound Tablets

In order to facilitate comparison of K-150, sugar-water, molasses-water and starch-water bound A·100W tablets, selected sintering data are shown in Table XV. Data were selected from sintering tests conducted at 930°F and 60 minutes except for tablets formulated with K-150 pitch binder in which the minimum temperature used was 1110°F. On the basis of sintering losses and residual hydrogen alone, these data indicate no pronounced advantages of using either sugar, starch, or molasses binder. However, as indicated earlier, utilization of K-150 pitch is not advisable. The data show that starch, sugar, and molasses bound A·100W tablets sustain significantly higher boron losses than do K-150 bound tablets. The fact that boric acid can be entrained by steam (supplied by water contained in the binder) accounts for this difference.

2. Boron, Carbon, and Hydrogen Losses for Boric Acid-Carbon Briquets

a. Starch-Water Bound Briquets

Table XVI summarizes the results of sintering experiments on A·100W briquets with a starch-water binder. These data are plotted in Figure 15 to show the effect of sintering time and temperature on sintering losses and residual hydrogen content. A comparison of sintering losses for A·100SWa briquets and tablets is shown below for sintering test conditions of 930°F and 1 hour:

Form	Formulation	Batch No.	Sintering Losses Per Cent				Residual H per cent
			overall	B	C	H	
Tablets	A·100W25SWa (80Wa)	54	45	19	-4	96	0.26
Briquets	A·100W·25.4SWa (88Wa)	46	48	15	10	98.5	0.12

In spite of the larger cross section of the briquets, the per cent hydrogen retained by the briquets after sintering was no greater than that retained by tablets thus indicating that briquets are equally amenable to sintering.

b. Sugar-Water Bound Briquets

Sintering data for A·100W briquets with a 20 per cent sugar-water binder are presented in Table XVII and Figure 16. The Cenco-Cooley muffle furnace operated by the prepilot group was used for these tests. Sintering loss data for both tablets and briquets are presented in the following table for comparison. Comparative data are for 930°F and 60 minutes residence time.

Form	Formulation	Batch No.	Sintering Losses Per Cent over-all	B	C	H	Residual Hydrogen Per cent
Tablets	A·100W·20SuWa (60Wa)	98	42	13	2	96	0.27
Briquets	A·100W·20SuWa (60Wa)	99	42	11	-1.6	98	0.2

As was in the case of A·100W starch-water formulations these data show that boric oxide-carbon briquet formulations prepared with sugar-water as binder sinter as well as tablets. Furthermore, there appears to be no difference in sintering losses between tablets and briquets. Approximately 16,000 pounds of sintered A·150W25SuWa (70Wa) briquets were prepared for pilot plant chlorination operations.

For the majority of these production tests, the following sintering cycle was used: (a) 1/2 hour hold time at each of the following temperatures: 400, 500 and 600°F; (b) 2 hours at 700°F; followed by (c) 1 hour at 900°F. A nitrogen purge was maintained during heating and cooling. When the temperature in the working chamber cooled to 700°F, the oven doors were opened to facilitate cooling. With the foregoing sintering procedure, a complete sintering cycle including allowance for loading and discharge time, required approximately 8 hours; a charge of 600 lb. of green briquets (200 lb./cycle) yielded 340 lb. of sintered feed daily.

Average sintering losses and residual hydrogen contents were as follows: 15 per cent boron; 2 per cent carbon; 43.5 per cent over-all; 0.35 per cent residual hydrogen. It is believed that the relatively high residual hydrogen content was the result of hydrogen picked up during preparation of the sample. Several production tests were performed in which the maximum temperature attained during the sintering cycle was only 800°F. In spite of this lower sintering temperature the hydrogen content of the sintered briquets was only 0.27 per cent as compared with an average of 0.30 per cent for three prior tests at 900°F. Production batches of boric oxide-carbon briquets were hard, undeformed and resistant to chipping and dusting.

3. Boron, Carbon and Hydrogen Losses for Boric Oxide-Carbon Granules

Sintering data for boric oxide-carbon granules with either water or sugar-water binder solutions are summarized in Tables VIII, IX, X and XVIII. A comparison of average over-all, boron, and carbon losses

and residual hydrogen content for 3/8 to 1/2 inch diameter granules versus A·100W tablets is presented below:

Form	Formulation	Sint. Temp °F	Losses, per cent Over-all	B	C	H	Residual Hydrogen Per cent
Tablets	A·100W20SuWa(80Wa)	930	41	15.7	7	99	0.26
Granules	A·100W·28.6Wa	1000	48.5	20.1	18.9	94.5	0.44

Higher average over-all losses for A·100W granules was attributed to higher boron and carbon losses in addition to the greater binder content. The increased binder content resulted in greater boron losses for granular feed by steam distillation of boric acid during sintering. A significant difference exists between granular and tabletted feed with respect to carbon losses which were 18.9 and 7 per cent respectively. This difference cannot be attributed to the increased oxidation due to higher sintering temperatures; reference to Figures 12A, C and D (for A·100W tablets) shows that at 1110°F the carbon loss was approximately 10 per cent. It is believed that the excessive carbon losses were the result of physical entrainment of carbon by the rapid evolution of water during sintering and/or dusting of the feed.

4. Boron, Carbon and Hydrogen Losses for Borax-Carbon Granules

Sintering data summarized in Table XIX emphasize difficulties encountered during drying and sintering which were mentioned in Section IIIA, B, and C. If carbon and boron losses were of a negligible value, the boron-carbon ratio would be 0.515 for the formulation A·100W. Excessive carbon losses occurred at 900°F as indicated by boron to carbon ratios of 1.19 and 2.09 for 1 and 2 hours residence time respectively. The data show that 750°F is the optimum sintering temperature and that a residual hydrogen content of 0.2 to 0.3 per cent must be tolerated.

5. Carbon and Hydrogen Losses for Witco Carbon Tablets

Preliminary development studies showed that the residual hydrogen content of Witco carbon tablets prepared with K-150 pitch binder was approximately 0.70 per cent even for sintering temperatures as high as 1100°F. No improvement was experienced for formulations with starch-water solutions as the binder. However, the utilization of sugar-water binder showed that Witco carbon tablets of the formulation 67W·33SuWa (70Wa) could be sintered at temperatures as low as 750°F to yield sintered stock having a hydrogen content of 0.2 to 0.35 per cent. Results showing residual hydrogen content and over-all sintering losses for the aforementioned formulation at temperatures ranging from 750°F to 1290°F and for residence times of from 20 to 180 minutes are presented in Table XX and illustrated in Figure 17. Increasing the sintering temperature much in excess of 750°F resulted in excessive carbon losses. The carbon loss under sintering conditions of 1 hour at 750°F was approximately 7 per cent.

6. Carbon and Hydrogen Losses for Witco Carbon Briquets

Carbon base briquets prepared with sugar-water binder of the nominal formulation 59W·41SuWa (63.6Wa) were sintered by the prepilot group without any operating difficulties. One-pound batches of unsintered briquets sintered at 1000°F exhibited sintering characteristics and losses essentially identical with those of 67W·33SuWa (70Wa) tablets except that higher over-all losses prevailed as a result of the increased binder content. However, when carbon briquets were sintered in the pilot plant Gehnrich batch oven (200 lb. capacity per batch) severe burning was observed even for temperatures as low as 300°F during the cooling cycle. As long as 48 hours was required to complete a sintering cycle, increasing the nitrogen purge from 0.5 to 1.0 cu. ft./hr. at S. T. P. had a negligible effect on the excessive burning. Replacing the oven door gasket and sealing small leaks, detected after a careful oven inspection, also failed to effect an improvement. Reducing the maximum sintering temperature to 600°F and the oven charge from 200 lb. to 100 lb. also was unsuccessful.

Attempts to suppress the excessive burning by sintering carbon and boric acid-carbon briquets at the same time proved to be successful. Two modifications of this technique were tried. In the first, 4 trays of boric acid-carbon briquets (25 lb./tray) were placed in the middle racks of the oven while the other two top and bottom trays contained carbon briquets. This technique was tried because it was observed that the middle trays in prior sintering tests exhibited the severest degree of burning. In the second modification of the "mixed sintering procedure" trays of carbon and boric acid-carbon briquets (4 each) were placed in the oven on alternate racks. The latter mixed sintering procedure was somewhat more effective than the former in reducing burning. Under these conditions, the sintering cycle was reduced from 48 to 20 hours, carbon losses were reduced significantly and the ratio of acceptable sintered briquets to total sintered material was approximately 0.9. It is believed that excessive burning may be attributed to (a) increased sugar required for briquetting and/or (b) the lower cooling rate of the Gehnrich pilot plant oven.

A comparison of sintering losses of carbon base tablets and briquets is presented below:

Form	Formulation	Sint. Temp. °F	Sintering Losses		Residual Hydrogen Per cent
			Over-all Per cent	Carbon Per cent	
Tablets	67W·33SuWa (70Wa)	750	33.0	7	0.28
Briquets*	59W·41SuWa (63.6 Wa)	800	47.7	13	0.35

* Alternate tray mixed sintering procedure

7. Effect of Predrying on Sintering Losses

a. Predrying of Boric Acid-Carbon Tablets

A study of the over-all sintering results suggested that carbon losses were primarily functions of time and temperature in excess of about 800°F whereas boron losses consistently ranged from 10 to 15 per cent irrespective of sintering time and temperature. Boron losses were attributed to steam distillation of boric acid occurring during the evolution and decomposition of binder and the dehydration of boric acid to boric oxide.

Table XXI (Batch 76) compares sintering losses for boric acid-carbon tablets not predried and predried at various temperatures ranging from 176 to 248°F prior to a 930°F sintering treatment. These data show that boron losses were reduced from 15.6 per cent for undried tablets to 12 per cent for tablets predried at 248°F. No change in hydrogen losses occurred. Table XXI (Batch 97) shows that increasing the predrying temperature to 350°F further reduced the boron losses to about 9 per cent.

In general, the data show that the drier the tablets were before the final sintering treatment, the lower the boron losses. Boron losses could not be reduced below 9 per cent.

b. Predrying of Boric Acid-Carbon Granules

Predrying tests were also performed on boric acid-carbon granules of several size fractions. Results from these experiments are shown in Tables XVIII and XXII. For convenience, a comparison of sintering losses for "regular" and predrying procedure is presented in Table XXIII. The data in Table XXIII indicate that predrying the granules for 1 hour at 200°F prior to the 1-hour, 1000°F sintering treatment appreciably reduced the boron losses and also reduced the carbon losses, but to a somewhat lesser extent.

8. Vacuum Drying Tests on Boric Acid-Carbon Granules

Vacuum drying tests with the use of a Precision Scientific Co. laboratory vacuum oven were performed to determine whether improvements could be obtained with respect to residual hydrogen content. Tests were conducted for varying drying times at temperatures ranging from 138°F to 415°F with 1/4 - to 3/8-inch diameter A·100W granules. A vacuum of 23 to 25 inches of mercury was maintained during the test period and data from this series of experiments are summarized in Table XXIV. These data indicate that an oven temperature between 194 and 260°F is required to remove essentially all of the free moisture and that some temperature in excess of 405°F is needed to dehydrate boric acid.

9. Effect of Sintering Methods on Degradation of Granular Feed

Results of a series of tests to determine the effect of various drying and sintering procedures on degradation of A·100W granules

are shown in Table XXV. Each of the three batches of granular feed which had been prepared under slightly different conditions was sintered as follows:

(a) 1 hour at 200°F plus heat up to 1000°F plus 1 hour at 1000°F

(b) 1 hour at 1000°F

(c) 1 hour at 200°F in drying oven and transfer to sintering oven at 1000°F for 1 additional hour

The amount of degradation was determined by screening through a 6 mesh sieve. Data obtained are rather difficult to explain in that the greatest degradation of particles occurred with the test procedure consisting of two-stage drying. Material in the trays was not disturbed between sintering stages. The results were in conflict with previous data that predrying was desirable both from the standpoint of reducing degradation of particles and boron and carbon sintering losses. A possible explanation of the excessive amount of degradation in the third treatment is that exposure at 200°F resulted in the formation of a hard surface on the granule which curtailed the evolution of vapors when it was exposed to 1000°F.

10. Oxidation Tests on Boric Acid-Carbon Granules

A series of 15 experiments were performed to determine boron and carbon losses of solid feed material under conditions similar to those encountered in commercial tunnel driers. The apparatus used for these experiments is shown in Figure 18. The test procedure consisted of passing preheated mixtures of nitrogen, oxygen, carbon dioxide, and water vapor through a 3 inch high vertical bed of unsintered, No. 6 mesh, 1/4-inch granules contained in a 1 inch inside diameter stainless steel pipe. In all tests, gases were fed at a total rate of 150 cu. ft. (S. T. P.) min/ft.^{2} of bed at temperatures of 800, 1000 and 1200°F for 1 or 2 hours duration. Operating conditions and results of the tests are summarized in Table XXVI. Data indicated that considerable carbon losses, presumably due to entrainment, were sustained even with usage of non oxidizing gases. With use of wet air or flue gas containing 4 per cent free oxygen, the carbon loss due to oxidation was apparently significant at bed temperatures in excess of 840°F (inlet gas temperature = 1000°F) and exceeded 50 per cent at bed temperatures of 1100°F. Also, boron losses appeared to be independent of temperature or oxidizing nature of the gas; but they were significantly affected by the humidity of the gas stream.

11. Rotary Calcination of Briquetted Feed

A series of seven experiments were conducted to determine the operating characteristics of a rotary calciner type drier for continuous drying of boric acid-carbon and carbon base briquets. Figure 19 shows the rotary calciner and allied components. Experimental data and results are presented in Table XXVII. Preliminary tests showed that a calciner speed of 1.2 r.p.m. and a slope of 0.28 inches per foot were required to give a retention time of approximately 20 minutes in the furnace zone and an overall residence time of 1 hour. For a calciner speed of 1.2 r.p.m. and a green

briquet feed rate of about 12 lb./hr. a slope of less than 0.28 inch per foot caused backup of material into the overflow port at the feed end of the cylinder. Although excessive briquet breakage was observed during feeding, 50 per cent for carbon and 30 per cent for boric acid-carbon, proper design of the feeding mechanism in a scale up would reduce breakage to a much lower level.

a. Rotary Calcination of Witco Carbon Briquets

In spite of the higher calciner chamber temperature for experiment C-4, 1380°F, as opposed to 1200°F for experiment C-2, the hydrogen content of C-4 calcined product was considerably higher. This difference was most likely the result of greater residence time for experiment FC-2, 0.4 hours as opposed to 0.2 hours for FC-4. In order to insure that the residual hydrogen content does not exceed approximately 0.3 per cent, it is believed that the residence time should be of the order of 0.5 hour. A comparison of sintering losses for carbon base briquets for regular drying and rotary calcination operations is presented below:

	Sintering		Sintering Losses		Residual
Formulation	Procedure	Temp. °F	Over-all Per cent	Carbon Per cent	Hydrogen Per cent
59W·41SuWa (63.6Wa)	Regular	800	47.7	13	0.35
59W·41SuWa (63.6Wa)	Rotary calcination	1290	39.5	13	0.39 to 1.83

b. Rotary Calcination of Boric Acid-Carbon Briquets

In experiment C-6 and C-7, backup of vent gases and resultant condensation of vapors due to a partial plug in the solids knockout box caused incoming feed to get wet and sticky. This, in turn, resulted in erratic flow of briquets as well as some fusion and incomplete sintering. Severe corrosion of the mild steel box and baffles (see Figure 19) was observed. A comparison of sintering characteristics of calcined briquets with those of the regularly sintered briquets is as follows:

	Sintering		Sintering Losses			Residual
Formulation	Procedure	Temp. °F	Over-all Per cent	B Per cent	C Per cent	Hydrogen Per cent
A·150W·25SuWa(70Wa)	Regular (pilot plant data)	900	43.5	15	2	0.35
A·150W·25SuWa(70Wa)	Calcined	1188	44.1	24	19.7	0.71 to 3.0

The excessive residual hydrogen content for calcined briquets may be the result of inadequate residence time in the furnace zone. Increasing the residence time from 0.2 hours to 0.27 hours (experiment C-5) was accompanied by a reduction in the residual hydrogen content to 0.35 per cent. A residence time of approximately 0.5 hours is recommended to insure elimination of hydrogen to the 0.2 to 0.3 per cent level.

The sharp increase in the carbon loss for rotary calcination experiments was primarily the result of higher sintering temperature and to a lesser extent the result of the formation of carbon dust which is more easily oxidized.

In view of the operating difficulties experienced during calcination tests with boric acid-Witco carbon feed, which are caused by the tendency of mixtures of boric acid and carbon to fuse, additional experimentation should be conducted to establish operating limitations and design characteristics. Use of a rotary calciner to sinter carbon base feed appears to be a feasible process-wise.

E. Vendor Tests

Selection of final equipment for tonnage feed compacting, drying, and sintering of boric acid-carbon pellets posed a problem due to the number and variety of equipment offered by industry. Therefore, several well-established companies known to have testing facilities for material evaluation were contacted and tests were arranged.

1. Dravo Corporation Disc Pelletizer

Inasmuch as laboratory tests had shown the feasibility of preparing spherical boric acid-carbon pellets which were satisfactory from the stand-point of reactivity (1, 2) as well as drying characteristics, investigation was made of commercially available equipment capable of producing tonnage quantities of the pellets.

A disc pelletizer offered by the Dravo Corporation of Pittsburgh, Pennsylvania, was tested. The Dravo pelletizer is a pan which rotates on a tilted axis. As the powdered mixture is fed to the disc, it is sprayed with water. The action of the rotating disc has a classifying effect and produces segregation of the pellets as they are produced. The larger pellets roll toward the rim of the disc and the smaller ones stay near the center. When the disc is full, there is a continuous overflow of the larger pellets. The action produces a uniform pellet size and eliminates the need for screening.

At the Dravo research laboratories, tests were conducted with use of a 39-inch diameter pan equipped with variable depth retaining walls and scraper blades. Pan speed and tilt were both variable. Feed material consisting of premixed powdered boric acid and lamp black in the ratio of 1.72:1 was fed at desired rates through a hopper-screw feeder arrangement detached from the pan. Water was metered through a flat-spray nozzle to the rolling bed. The tests were performed at pan speeds ranging from 25 to 30 r.p.m. and pan inclinations from 42 to 48 degrees. Control of solids retention time by pan tilt, feed rate and retainer depth yielded spherical nodules of any size. Pellets were uniform in size, shape and surface characteristics. Test data indicated that boric acid-carbon pellets of about 3/8 inch diameter could be produced at the rate of 28 lb./hr./ft.^{2} of pan area or about 244 lb./hr. with the 39-inch pan. Equipment can be obtained in pan diameters up to 16 feet.

A thirty-nine inch diameter Dravo-Lurgi Pelletizing Disc was rented from the Dravo Corporation and a limited number of experiments were conducted in the pilot plant. Approximately 866 lb. of unsintered 1/2 inch + pellets were prepared with no operating difficulties.

2. Link Belt Co. Slurry Nodulation and Drying Tests

In January, 1958, bench tests were conducted at the Link-Belt Material Handling and Testing Laboratory in Chicago. The aim of the tests was: (a) to determine feasibility of preparing homogeneous pellets of boric acid and F-1 Witco carbon by slurry nodulization and (b) to obtain drying characteristics of the boric acid-carbon pellets in a Link-Belt Roto-Louvre dryer. Bench equipment used for the tests consisted of a 2 cubic foot can, a rotary drum and a 1 gallon capacity Roto-Louvre dryer. It was anticipated that scale-up tests if warranted by results of the bench tests would be conducted in their 5 ton/hr. pilot plant consisting of a pug-mill, dryer, and cooler.

The following assumptions were used as a basis for formulation of mixtures:

1. A 5-10 per cent boric acid solution at approximately 180°F would be available as recycle from the dryer vent scrubber.

2. The amount of boric acid (in solution) available as recycle would be equivalent to 10 per cent of the solid acid feed.

3. The crushed solids recycle from the dryer screen would amount to 40 per cent of fresh feed.

Results of the slurry nodulization tests indicated that homogeneous pellets of boric acid and carbon can be prepared by gradual addition of solids to a hot boric acid solution. However, control of pellet size is difficult. Furthermore, it was found that the concentration of the slurry produced a viscous mixture which was difficult to maintain in a fluid state preparatory to balling. It is anticipated that power requirements for a mixer in this type of process would be high.

Results of drying tests conducted with the Roto-Louvre Dryer on wet pellets prepared by slurry nodulization are shown in Figures 20 and 21. Three drying tests were conducted at Link Belt with 53 cu. ft./min. inlet air for drying. Evolution of fumes, presumably boric acid was observed almost immediately after introduction of solids. In addition, a slight amount of carbon black was observed issuing from the drier exhaust port throughout each test. The granular feed exhibited a tendency to stick at material temperatures between 250 and 300°F in test R-2 and 296 to 496°F in test R-1. However, at higher down-stream temperatures the agglomerated particles disintegrated. The tests at Link Belt Co. showed that granular feed consisting of mixtures of boric acid and Witco F-1 could be dried from 22 per cent free moisture to 10 per cent in approximately 12 minutes at 215°F material temperature and to 5 per cent free moisture in approximately 19-1/2 minutes at 230°F material temperature with 53 cu. ft./min. inlet air at 900°-980°F. With the use of the data of Figure 20, the nominal drying rate to zero per cent free moisture was approximately 0.37 lb. H_2O/hr./lb. dry material.

3. General American Transportation Corp. Two-Stage Drying Tests

Tests were performed at the laboratory of the General American Transportation Corporation (GATX) at East Chicago, Indiana to determine the feasibility of a two-stage rotary drying unit to prepare granular reactor feed material. Seven predrying tests were conducted with a direct-fired, 1 ft. diameter by 6 ft. long flighted rotary drum at various gas temperatures from 600°F to 1100°F. With predried solids from the foregoing tests, two second stage drying experiments were performed in an indirect-fired, 1 ft.diameter by 8 ft. long rotary drum in which the calcined (dried) feed attained temperatures of 1060 to 1080°F. Operating conditions and results from the GATX two stage drying tests are presented in Table XXVIII.

A layer of solid material 18 inches long by 3/4 inch thick coated the first stage drier. In the second stage drier the same sticking tendency was observed. Excessive cohesion between granules as well as some fracturing of individual granules was observed during test 1 FD-2 in which the raw (predried feed stock from test 7) feed was obtained under the best conditions for predrying. The product was definitely not suitable for use as reactor (chlorinator) feed material. These tests showed that two stage drying as performed by GATX was not suitable for preparation of reactor feed material.

4. Proctor and Schwartz Corp. Drying Tests

Drying tests on boric acid-Witco F-1 carbon granules were conducted in the laboratories of the Proctor and Schwartz Corporation, Philadelphia Pennsylvania, with the use of a single tray batch through-circulation dryer. The unsintered granules were contained in a one-foot by one-foot perforated bottom tray and loaded to a depth of 1-1/2 inches. Air at several wet and dry bulb temperatures was passed through the bed of granules at a constant rate of 250 ft./min. superficial velocity. Dried material was forwarded to Olin Mathieson laboratories at Niagara Falls for sintering tests at 1000°F to permit comparison of hardness and stability. Table XXIX summarizes the results of the tests at Proctor and Schwartz and subsequent sintering tests at Niagara Falls.

Table XXIX shows that all of the free moisture was removed during each drying test and that some of the orthoboric acid, H_3BO_3, had decomposed to the meta form, HBO_2, liberating additional water. The dried material from all of the drying tests showed white deposits of boric acid on the surface of the granules. There was considerable spalling and cracking of the granules during the high temperature tests (No. 2, 4 and 5). The higher humidities in tests 4 and 5 resulted in increased boric acid vaporization (boron losses). This vapor escaped through leaks in the drier and deposited as a white solid on the external surfaces of the equipment. From a standpoint of boron losses and dried particle characteristics, tests No. 1 and 3 produced superior dried granules. The best sintered product obtained was with use of material from these same p and s runs tests (1 and 3). These products compared favorably with that obtained under standard laboratory conditions. The data summarized in Table XXIX show that operation of a perforated tray through circulation batch type dryer at 250 ft./min. of air

heated to 200°F dry bulb temperature and 100°F wet bulb temperature produced physically acceptable granular feed containing no free moisture and sustaining boron and carbon losses of only 4.6 and 4.3 per cent respectively. The drying data of test No. 1 yielded a nominal drying rate of 0.6 lb. H_2O/hr./lb. dry material.

F. Handling and Storage

1. Hydroscopicity of Sintered Briquets

Unaccountable variations in residual hydrogen contents of sintered pellets had been attributed to absorption of moisture during handling and storage. To determine whether this assumption was justifiable, moisture absorption tests were conducted on several samples of sintered briquets varying in composition. The samples were exposed to various temperature and humidity conditions and weight gains were recorded at several time intervals. Results from these tests are shown in Figure 22. The conditions selected were typical summer day conditions: temperatures ranging from 75°F to 90°F and relative humidities ranging from 31 to 62 per cent.

Figures 22A and 22B show that the rate of absorption was approximately twice as fast for A-100W as for A-50W briquets, and for both formulations, the rate of absorption was about twice as fast at 83°F and 62 per cent relative humidity as it was at 75°F and 31 to 38 per cent relative humidity. Figure 22C for A-200W and A-300W briquets substantiates the former data and emphasizes that increasing the carbon to boron ratio promotes higher absorption rates. The A-300W sample absorbed 7.4 per cent moisture in 8 hours and 13 per cent in 30 hours at 90°F and 48 to 58 per cent relative humidity. These data show that feed briquets are quite hygroscopic and that proper precautions must be taken for storage and handling.

2. Bulk Density of Sintered Briquets

The effect of increasing the carbon content of feed briquets on bulk density of the sintered briquets is shown in Figure 23. The densities range from 23 to 32 pounds per cubic foot as indicated by the data for A-100 briquets; resintering of the briquets increased the bulk density.

IV. CONCLUSIONS

1. Porous, hard, boric oxide-carbon tablets, or almond shaped briquets, may be prepared by compacting a mixture of 60 or 100 mesh boric acid powder, Witco carbon SRF beads or F-1 powder, and sugar-water binder and then sintering the green material at 800 to 900°F for 1 hour. Sintering losses of 15 per cent boron, 7 per cent carbon and a residual hydrogen content of 0.2 to 0.3 per cent are to be expected.

a. Predrying the material at 350°F prior to the 800-900°F finishing treatment reduces the boron losses to about 9 per cent but reduces the carbon losses only slightly.

b. The use of higher sintering temperatures fails to effect a significant reduction in the residual hydrogen content and results in higher carbon losses due to oxidation.

c. Substitution of K-150 petroleum pitch, starch-water, molasses-water or brown sugar-water as binder, and petroleum coke for Witco furnace black, is uneconomical or adversely affects sintered feed chemical and/or physical characteristics.

2. Sintered boric oxide-carbon granules, 1/4 - to 1/2-inch diameter containing 6 moles carbon per mole of boric oxide, (A-100W), may be prepared by spraying a stream of water into a bed of boric acid and carbon contained in a rotary drum granulator and then sintering the resultant granules under conditions identical with those employed for tabletted and briquetted feed. Sugar is not needed to yield satisfactory granules.

3. The preparation of carbon tablets and briquets can be performed in the same manner as used for boric acid-carbon tablets and briquets but binder requirements are greater due to the absence of boric acid. Sintering conditions of 750°F for 1 hour are required, resulting in a carbon loss of 7 per cent and a residual hydrogen content of 0.28 per cent. A nitrogen purge should be maintained during sintering and the briquets should be quick-chilled in a nitrogen atmosphere to prevent burning.

4. The rotary granulation method is not adaptable to the preparation of carbon granule feed because of excessive dusting and cracking incurred on sintering.

5. Difficulty with regard to control of granule size and anticipated high power requirement due to the slurry viscosity may preclude slurry granulation on a commercial scale. Although a Link Belt Roto-Louvre dryer successfully reduced the free moisture content of wet granules to 5 or 10 per cent, the heat load (inlet gas temperature 900 to 980°F) is a factor which prevents serious consideration of this system.

6. A disc pelletizer offered by the Dravo Corporation may be used to prepare boric acid-carbon granular feed.

7. Design of large-scale drying (sintering) equipment should include appropriate accessory equipment for predrying to minimize boron losses.

a. The use of a two-stage rotary dryer to predry and sinter boric acid-carbon granules is feasible provided that the rate of binder and water evolution is not excessive.

b. A perforated tray through circulation batch-type dryer requiring air heated to only 200°F produced physically acceptable pre-dried granules containing zero per cent free moisture.

8. Hygroscopicity tests on sintered boric oxide-carbon briquets showed that considerable moisture pickup occurs at typical atmospheric temperature and humidity conditions. Sintered feed material should be stored in airtight containers or in containers provided with a dry-nitrogen purge.

9. The following system is suggested for the preparation of boric oxide-carbon compacted feed on a large scale.

a. Premix boric acid and carbon in a ribbon mixer.

b. Add water (no sugar) to the premixed dry ingredients contained in a rotary granulator.

c. Predry and sinter in a two-stage rotary dryer at 356° and 800-900°F material temperatures.

10. The following system is suggested for the preparation of carbon compacted feed on a large scale.

a. Premix carbon and sugar and add water to mix contained in a ribbon mixer.

b. Press the damp feed into briquets.

c. Sinter the briquets in a tunnel dryer at a material temperature of 750°F.

V. LITERATURE REFERENCES

1. Caro, P. Olin Mathieson Chemical Corporation, Technical Report OMCC-HEF-100, March 31, 1958.

2. Derrick, G. C., Olin Mathieson Chemical Corporation, Technical Report OMCC-HEF-158, March 11, 1959.

VI. ATTACHMENTS

Table I to XXIX inclusive

Figure 1 to 23 inclusive

TABLE I
SUMMARY OF TABLET DEVELOPMENT EXPERIMENTS

Boron Carrier	Carbon Carrier	Binder	Optimum Formulation	Tabletting Characteristics: Drop Test Inches	Tabletting Characteristics: Remarks	Sintering Characteristics: Temp °F (a)	Sintering Characteristics: Remarks
1. Boric Oxide Base Tablets							
B_2O_3	Witco	No. 600 oil	O·20W·3 (6-oil)	18	Weak Pellets	1110	Fused. Soft at Red Heat
B_2O_3	Witco	No. 600 oil	O·100W·3 (6-oil)	24	Good Pellets	1110	Fused Slightly
B_2O_3	Witco	No. 600 oil	O·150W·3 (6-oil)	24	Good Pellets	1110	No fusing. Separates easily
B_2O_3	Witco	K-150	O·20W·20K150	24	Good Pellets	1110	Separates Easily. Hard Pellets
B_2O_3	Witco	K-150	O·100W·25K150	27	Good Pellets	1110	Separates Easily. Hard Pellets
B_2O_3	Witco	K-150	O·200W·25K150	18	Good Pellets	1110	Separates Easily. Hard Pellets
B_2O_3	Witco	K-200	O·10W·30K200	16	Poor Pellets	1110	Fusing. Pellets Weak
B_2O_3	Witco	K-200	O·20W·20K200	20	Good Pellets, but soft	1110	Slight fusing
B_2O_3	Witco	K-200	O·100W·25K200	21	Good Pellets	1110	No fusing. Pellets soft at red heat
B_2O_3	Witco	K-200	O·150W·25K200	22	Good Pellets	1110	No fusing. Hard at red heat
2. Boric Acid Base Tablets							
H_3BO_3	Witco	K-150	A·50W·15K150	Not done	Good Pellets	1020	Severe fusing
H_3BO_3	Witco	K-150	A·100W·25K150	15	Good Pellets	930	Severe fusing
H_3BO_3	Witco	K-150	A·500W·25K150	12	Poor Pellets	1020	Slight fusing
H_3BO_3	Witco	K-150	A·1000W·25K150	24	Poor Pellets	1020	Some fusing
H_3BO_3	Witco	K-150	A·1000W·25K150	24	Poor Pellets	1110	Severe fusing
H_3BO_3	Witco	K-150	A·1000W·25K150	24	Poor Pellets	1290	Completely fused
H_3BO_3	GLC (b)	K-150	A·20GLC·8K150	1444	Good, very hard Pellets	930	Fusing and swelling
H_3BO_3	GLC	Water	A·25GLC·4Wa	1444		930	Fusing and swelling
H_3BO_3	Witco	SWa	A·100W·25SWa(70Wa)	28	Fair Pellets	750	Slight fusing
H_3BO_3	Witco	SWa	A·200W·25SWa (70Wa)	12	Poor Pellets	930	Slight fusing
H_3BO_3	Witco	SWa	A·500W·33SWa(70Wa)	No Record		1290	----------
H_3BO_3	Witco	SWa	A·1000W·25SWa(70Wa)	No Record		1290	----------

(a) Where only one temperature is presented it is the maximum temperature to which the pellets were subjected.

(b) Great Lakes Carbon (Powder)

TABLE I

SUMMARY OF TABLET DEVELOPMENT EXPERIMENTS (Continued)

Boron Carrier	Carbon Carrier	Binder	Optimum Formulation	Tabletting Characteristics: Drop Test Inches	Tabletting Characteristics: Remarks	Sintering Characteristics: Temp °F (a)	Sintering Characteristics: Remarks
2. Boric Acid Base Tablets (con't.)							
H_3BO_3	Witco	SuWa	A·50W·15SuWa(80Wa)	36+	Good Pellets	1110	Severe fusing
H_3BO_3	Witco	SuWa	A·100W·20SuWa(80Wa)	36+	Good Pellets	1290	No fusing or deformation
H_3BO_3	Witco	SuWa	A·200W·20SuWa(80Wa)	48+	Good Pellets	1290	No fusing or deformation
H_3BO_3	Witco	SuWa	A·300W·25SuWa(80Wa)	48+	Good Pellets	1290	No fusing or deformation
H_3BO_3	Witco	SuWa	A·500W·25SuWa(70Wa)	48+	Good Pellets	1290	No fusing or deformation
H_3BO_3	Witco	BSuWa	A·100W·20BsuWa(60Wa)	120	Good Pellets	1100	No fusing or deformation
H_3BO_3	Beaded	SuWa	A·50WSRF·15SuWa(80Wa)	N. R.	No Record	1000	Severe fusing
H_3BO_3	Witco SRF	SuWa	A·100WSRF·20SuWa(80Wa)	N. R.	No Record	1000	Severe fusing
H_3BO_3	Pet. Coke	SuWa	A·100PC(SuWa)	N. R.	Good Pellets	1000	Severe fusing
H_3BO_3	Pet. Coke	SuWa	A·200PC(SuWa)	N. R.	Good Pellets	1000	Severe fusing
H_3BO_3	Calcined Gilsonite	Su only	A·100CG·10Su	N. R.	Poor	1000	Severe fusing
H_3BO_3	Witco	Wa only	A·100W·XWa	N. R.	Poor Green Strength	1000	No fusing. Excellent strength
H_3BO_3	Witco	MWa	A·100W·20MWa(60Wa)	N. R.	Good	1000	No fusing
3. Borax Base Tablets							
Razorite ($Na_2B_4O_7$)	Witco	SuWa	NBO·100W·45SuWa(80Wa)	N. R.	Good	1000	Severe fusing
$Na_2B_4O_7$ (Anhydrous)	Witco	SuWa	NBO·100W·45SuWa(80Wa)	N. R.	Good	1000	Little fusing
4. Carbon Base Tablets							
----	Witco	K-150	85W·15K150	12	Poor Pellets fluffy mix	1290	No fusing; but considerable cracking + dusting. Pellets weak.
----	Witco	SWa	67W·33Swa(70Wa)	18	Fair Pellets	1290	No fusing; Physical characteristics better than 85W 15K150
----	Witco	SuWa	67W·33SuWa(70Wa)	18-24	Fair Pellets	1290	No fusing; Physical characteristics improved but inferior to boron base stock

(a) Where only one temperature is presented it is the maximum temperature to which the pellets were subjected.

N. R. = No Record

TABLE II
SUMMARY OF PELLET TESTS USING BORIC OXIDE AND WITCO BLACK WITH VARIOUS BINDERS AND BORIC OXIDE/CARBON RATIOS

Test No.	Excess Carbon Per cent	Binder	Binder Per cent	Length inches	Drop Test	Condition of Pellet at Sintering Temp. (a)	Comments on Preparation of Pellet, Strength, Etc.	Comments on Pellet Condition After Sintering (b)
1	20	No. 600 Oil	3	24/128	18	Very Soft	Weak Pellet	Fused together, deformed with pressure
2	100	No. 600 Oil	3	26/128	24	Fairly Soft	Good. Free Flowing Mix	Fused Slightly
3	100	No. 600 Oil	6	24/128	21-24	Slightly Soft	Soft, Cracked. Not Free-flowing	Sticky when hot
4	100	No. 600 Oil	12			Slightly Soft	Doesn't Flow	Sticky when hot
5	100	No. 600 Oil	18			Slightly Soft	Mix not free-flowing	Separates easily
6	150	No. 600 Oil	3	25/128	24	Fairly Soft	Soft. Free-flowing mix	Separates easily
7	150	No. 600 Oil	6	25/128	24	Fairly Soft	Soft. Free-flowing mix	Separates easily
8	150	No. 600 Oil	9	28/128	18	Slightly Soft	Cracked Badly	Separates easily
9	20	K-150	15	27/128	20	Slightly Soft	Free-flowing, soft	Separates easily
10	20	K-150	20	28/128	24	Fairly Hard	Free-flowing,	Separates easily
11	50	K-150	25	28/128	33	Slightly Soft	Free-flowing, Good Pellet	Not sticky at red heat
12	100	K-150	25	25/128	27	Slightly Soft	Good	Not sticky at red heat, separates easily
13	150	K-150	25	25/128	27	Hard	Good	Separated easily. Deforme with pressure
14	200	K-150	20	28/128	12	Soft	Free-flowing. Soft Pellet	Separated easily. Deforme with pressure
15	200	K-150	25	27/128	18	Soft	Difficult to Mix Soft Pellet (c)	Separated easily
16	200	K-150	30	28/128	19	Fairly Hard	Not free-flowing. Lumpy	Separated easily
17	200	K-150	35	---	21	Fairly Hard	Not free-flowing. Lumpy	Separated easily
18	200	K-150	40	27/128	24	Fairly Hard	Not free-flowing. Lumpy	Stuck together
19	10	K-200	30	27/128	16	Weak	Weak	Fused together slightly
20	20	K-200	20	24/128	18	Very Soft	Weak, free-flowing	Fused together slightly
21	20	K-200	25	25/128	20	Fairly Soft	Free-flowing	Not sticky at red heat
22	100	K-200	25	24/128	21	Fairly Soft	Free-flowing	Separated easily
23	100	K-200	30	28/128	15-21	Hard	Capped	Very slight fusing at red he
24	150	K-200	25	25/128	22	Hard	Free-flowing	
25	200	K-200	25	---	Low		Very weak	

(a) All pellets were 1/4 inch diameter and sintered at 600°C for 15-20 minutes.

(b) After sintering all the pellets tested were undeformed, and very hard after cooling.

(c) Characteristics of this pellet improved greatly when mixing thoroughly in Baker-Perkins Mixer.

TABLE III
SUMMARY OF BORON-CARBON PELLET PREPARATION EXPERIMENTS

	B Carrier	Excess Carbon (a) Per cent	Type	Binder (a) Per cent	Type	Pellet Diam. in.	Condition of Powder	Pellet Drop Test Inches	Per cent of Pellets Breaking on Ejection	Sintering Temp. °C	Condition After Sintering
Part of Statistical Program	H_3BO_3	5	WITCO	20	K-150	1/4	Free Flowing	> 144		500	Flowed, Swelle
	H_3BO_3	15	WITCO	4	K-150	1/4	Free Flowing	29		500	Flowed, Swelle
	H_3BO_3	15	WITCO	20	K-200	1/4	Free Flowing	12		500	Flowed, Swelle
	H_3BO_3	20	GLC	8	K-150	1/4	Free Flowing	>144		500	Flowed, Swelle
	H_3BO_3	25	GLC	4	Water	1/4	Lumpy, Not free flowing	>144		500	Flowed, Swelle
	H_3BO_3	25	WITCO	12	K-200	1/4		25		500	Flowed, Swelle
	H_3BO_3	10	WITCO	2	K-150	1/4	Free flowing		95	600	Soft, Fused and deformed
	H_3BO_3	10	WITCO	4	K-150	1/4	Free flowing		90	600	Soft, Fused and deformed
	H_3BO_3	10	WITCO	6	K-150	1/4	Free flowing		75	600	Soft, Fused and deformed
	H_3BO_3	10	WITCO	8	K-150	1/4	Free flowing		57	600	Soft, Fused and deformed
	H_3BO_3	10	WITCO	10	K-150	1/4	Free flowing		47	600	Soft, Fused and deformed
	H_3BO_3	10	WITCO	14	K-150	1/4	Free flowing		25	600	Soft
	H_3BO_3	10	WITCO	18	K-150	1/4	Free flowing		15	600	Fair
	H_3BO_3	10	WITCO	22	K-150	1/4	Free flowing		40	600	Very poor
	H_3BO_3	100	WITCO	30	W-S	1/4	Free flowing			700	Good
	H_3BO_3	200	WITCO	30	W-S	1/4	Free flowing			700	Good
	H_3BO_3	10	WITCO	0		1/4	Free flowing		15	600	Fused, Flowed
	H_3BO_3	10	WITCO	3	W-S	1/4	Free flowing		10	600	Fused, Flowed
	H_3BO_3	10	WITCO	6	W-S	1/4	Free flowing		8	600	Fused, Flowed
	H_3BO_3	10	WITCO	12	W-S	1/4	Free flowing		5	600	Fused, Flowed
	H_3BO_3	10	WITCO	20	W-S	1/4	Free flowing		0	600	Fused, Flowed
	H_3BO_3	10	WITCO	30	W-S	1/4	Free flowing		0	600	No deformation very slightly fused. Soft.

(a) List of Abbreviations

K-150 Koppers Pitch (Melt Pt. = 140-150°F)

K-200 Koppers Pitch (Melt Pt. = 200-220°F)

W-S 2/3 Potato Starch, 1/3 water mixture

GLC Great Lakes Carbon Petroleum Coke

WITCO WITCO Channel Black

TABLE IV
SUMMARY OF BRIQUET DEVELOPMENT EXPERIMENTS
BORIC ACID-SRF WITCO CARBON-STARCH WATER BRIQUETS

Expt. No.	Formulation	Green Briquets (a) S/(BA+W)	Green Briquets (a) Wa/(BA+W)	Green Briquets (a) Remarks (mixing and briquetting) (b)	Sintered Briquets Sintering Characteristics	Sintered Briquets Muffling Characteristics
3	A·100W·25SWa (88Wa)	0.04	0.294	Good Briquets	--	--
17	A·100W·38.4SWa (60Wa)	0.25	0.373	Good Briquets	--	--
30	A·100W·27.2SWa (89.5Wa)	0.04	0.344	Fair Briquets	Not Fused	Fused
31	A·100W·30.8SWa (77Wa)	0.1	0.344	Good Briquets	Not Fused	Fused
32	A·100W·30.8SWa (77Wa)	0.1	0.344	Good Briquets	Not Fused	Fused
34	A·100W·35.5SWa (82Wa)	0.1	0.45	Good Briquets	Not Fused	Fused
35	A·100W·37.5SWa (80Wa)	0.15	0.45	Good Briquets	Not Fused	Fused
40	A·100W·40.5SWa (70.5Wa)	0.20	0.48	Good Briquets	Not Fused	Fused
42	A·100W·42.9SWa (66.6Wa)	0.25	0.50	Good Briquets	Not Fused	Fused
44	A·100W·45SWa (63.5Wa)	0.3	0.52	Poor Briquets	Not Fused	Fused
47	A·100W·45SWa (57.3Wa)	0.35	0.47	Poor green strength	Not Fused	Fused
48	A·100W·25.4SWa (88Wa)	0.04	0.31	--	--	--
		Batches 21 and 24 through 29 used cooked starch				
21	A·100W·48.8SWa (89.5Wa)	0.1	0.854	Too wet. Not briquetted	--	--
24	A·100W·33SWa (80Wa)	0.1	0.4	Made 1/2" balls in mixer. Not briquetted	--	--
25A	A·100W·45SWa (88Wa)	0.1	0.75	Too w t	--	--
25B	A·100W·39SWa (88Wa)	0.075	0.563	Made balls, too wet	--	--
25C	A·100W·33.8SWa (88Wa)	0.06	0.451	Too wet	--	--
26	A·100W·24.4	0.06	0.234	Incomplete mixing balls of pure starch	--	--
27	A·100W·24.4SWa (87.5Wa)	0.04	0.28	Mix slightly dry good briquets	--	Not Fused
28	A·100W·27.7SWa (89.5Wa)	0.04	0.34	Started to ball-good briq.	--	Not Fused
29	A·100W·27SWa (86.6Wa)	0.05	0.32	Incomplete mixing fair briquets	--	Not Fused

(a) Density, green briquets = 45 lbs./ft.^{3}

(b) Drop test = (number of briquets unbroken number of briquets dropped) x 100. Six-foot drop onto plywood.

TABLE V

SUMMARY OF BRIQUET DEVELOPMENT EXPERIMENTS
BORIC ACID-SRF WITCO CARBON-SUGAR WATER BRIQUETS

Expt. No.	Formulation	Green Briquets					Sintered Briquets		
		Su/BA+W	Wa/BA+W	Density lb./ft.^{3}	Drop Test Per cent (a)	Remarks (mixing and briquetting)	Density lb./ft.^{3}	Sintering Characteristics	Muffling Characteristics
7	A-1000W					No briquets formed	--	--	--
8	A-100W					No briquets formed	--	--	--
9	A-100W-25Wa		0.332			No briquets formed	--	--	--
						Variation of Binder Content			
-	A-100W-16.7SuWa(80Wa)	0.040	.161			Too soft			
2	A-100W-25.5SuWa(85Wa)	0.054	.288	--	84	Good Briquets	33.4	Not Fused	Fused
11	A-100W-34SuWa(61.4Wa)	0.200	.316		80	Wet Mix Poor Feeding Good Briquets	33	Not Fused	Fused
12	A-100W-23.1SuWa(66Wa)	0.1	0.2			Damp Mix, Not briquetted	--	--	--
13	A-100W-20SuWa(60Wa)	0.1	0.15	50.6	100	Good Briquets	33.1	Not Fused	Fused
15	A-100W-25.9SuWa(42.9Wa)	0.2	0.15	50.9	100	Good Briquets	--	--	--
16	A-100W-31.1SuWa(33.3Wa)	0.3	0.15	49	--	Good Briquets	--	Not Fused	Not Fused
						Variation of Per cent Excess Carbon			
6	A-100W-20SuWa(80Wa)	0.05	0.20	51.5	70	Good Briquets	31	Fused	Fused
10	A-100W-25SuWa(70Wa)	0.10	0.21	--	88	Good Briquets	34	Slight Fusing	Fused
20	A-100W-35.5SuWa(27.3Wa)	0.4	0.15	--	--	Good Briquets	--	No Fusing	No Fusing
53	A-200W-34SuWa(41.2Wa)	0.3	0.21	--	--	Good Briquets	--	No Fusing	No Fusing
63	A-25W-20SuWa(60Wa)	0.1	0.15	--	--	Good Briquets	--	Fusing	Fusing
65	A-OW-15SuWa(60Wa)	0.07	0.11	--	--	Good Briquets	--	Fusing	Fusing
68	A-224W-32.8SuWa(41.2Wa)	0.287	0.20	--	--	Good Briquets	--	No Fusing	No Fusing
70	A-100W-30SuWa(30Wa)	0.3	0.13	--	--	Good Briquets	35.3	No Fusing	No Fusing
74	A-100W-30SuWa(30Wa)	0.3	0.13	--	--	Good Briquets	37.9	No Fusing	No Fusing
76	A-150W-18.6SuWa(56.5Wa)	0.1	0.13	--	--				No Fusing
81	A-100W-20SuWa(60Wa)	0.1	0.15	--	--	Good Briquets	28	No Fusing	Some Fusing
87	A-40W-21.5SuWa(62.8Wa)	0.1	0.17	--	--	Good Briquets	29	No Fusing	Some Fusing
89	A-40W-18.6SuWa(56.5Wa)	0.1	0.13	--	--	Good Briquets	31.8	--	Fused
92	A-40W-18.6SuWa(56.5Wa)	0.1	0.13	--	--	Good Briquets	30.2	--	Fused

(a) Drop Test = (number of briquets unbroken + number of briquets dropped) x 100.
The test is a 6 foot drop onto plywood

TABLE VI
SUMMARY OF BRIQUET DEVELOPMENT EXPERIMENTS
SRF WITCO CARBON-STARCH WATER BRIQUETS

Expt. No.	Formulation	Green Briquets			Sintered Briquets	
		S/W	Wa/W	Remarks (Mixing and Briquetting)	Sintering Characteristics	Muffling Characteristics
49	65W·35SWa(81.5Wa)	0.1	0.44	Good Briquets	Poor strength	Easily crush after muffling
50	52.9W·47.1SWa(77.5Wa)	0.2	0.69	Good Briquets	Poor strength	Easily crush after muffling
51	48.8W·51.2SWa(71.5Wa)	0.3	0.75	Weak Briquets	Poor strength	--
52	47.25W·52.75SWa(68.8Wa)	0.35	0.77	Very Weak Briquets	Poor strength broke up during sintering	--

TABLE VII
SUMMARY OF BRIQUET DEVELOPMENT EXPERIMENTS
SRF WITCO CARBON-SUGAR WATER BRIQUETS

Expt. No.	Formulation	Green Briquets			Sintered Briquets	
		Su/W	Wa/W	Remarks (mixing and briquetting)	Sintering Characteristics	Muffling Characteristics
19	69W·31SuWa(66.7Wa)	0.15	0.3	Very poor strength	Very poor strength	--
36	69W.31SuWa(33.3Wa)	0.3	0.15	Poor briquets	No fusing	--
37	72.7W·27.3SuWa(40Wa)	.225	0.15	Poor briquets	No fusing	--
38	62.5W·37.5SuWa(41.6Wa)	0.35	0.25	Good briquets	No fusing strong but brittle	No fusing. 1/2 to 1/3 broke during muffling
38A	58.8W·41.2SuWa(50Wa)	0.35	0.35	Good briquets	No fusing strong but brittle	No fusing. 1/2 to 1/3 broke during muffling
84A	57.2W·42.8SuWa(53.3Wa)	0.35	.296	Good briquets (mix was dry)	--	--
84B (a)	57.2W·42.8SuWa(53.3Wa)	0.35	.296	Good briquets (mix was dry)	--	--
--	59W.41SuWa(64Wa) (Optimum Formulation)	0.25	0.44	Good briquets	No fusing strong but brittle	No fusing. Some attrition during muffling

(a) Witco carbon in ribbon mixer was dry. Mixed prior to addition to blender.

TABLE VIII
SUMMARY OF GRANULATION EXPERIMENTS WITH A-100W-"X"SuWa FORMULATIONS

Expt. No.		G-12	G-13	G-14	G-15	G-16	G-17
"X"Su, Per cent		4.8	4.8	10	10	2	2
H_2O Added, Per cent of Charge		33.5	33	30.5	29	30.5	28
Moisture Content of Balls Per cent		18.8	20.5	20.0	17.4	15.8	17.0
Time for H_2O Addition, Minutes		10	15	20	15	10	15
Additional Time for Granulation, Minutes		32	30	30	35	35	35
Drying Loss, Per cent		14	8.5	not dried	10	12	11
Sintering Loss, Per cent		50	47	48	50	47	46
Screen Analysis of Sintered Balls, Per cent Retained On	3/4 in.	0	0	0	0	0	0
	1/2 in.	1.5	0	4.5	15	0	0
	1/4 in.	46	24.5	74.5	76.5	4	5.5
	No. 6	42	58	20.5	6.5	57.5	56
	No. 14	7	15.5	0.5	1.5	32	31
	Pan	3.5	2.0	0	0.5	6.5	7.5
	Total	100.0	100.0	100.0	100.0	100.0	100.0
Residual Hydrogen of Screened Fractions, Per cent		0.87		0.37	0.40		
		0.31	0.26	0.28	0.37	0.13	0.24
		0.33	0.27	0.31	0.44	0.53	0.19
		0.38	0.31	0.44	0.69	0.16	0.09
		0.50	0.76		1.60	0.13	0.19

TABLE IX

SUMMARY OF OPERATING DATA FOR GRANULATION EXPERIMENTS

Expt. No.	G-19 (a)	G-20	G-20RR (b)	G-21 (b)	G-22	G-23	G-23R	G-24	G-25RR	G-26	G-27 (d)	G-28 (d)	G-31 (d)
Dry Mix, g.					2268	2270	2270	2270	2268	2268	4305	4305	4305
Boric Acid, g.					1433	0	0	227	1433	1433	2470	2470	2470
Carbon, g.					835	2043	2043	2043	835	835	1440	1440	1440
Sugar, g.	4.8 (c)	0	0	0	0	227	227	0	0	0	395	395	395
Water Added, per cent	26.4	39.7	41.9	39.7	37.5	61.7	57.2	72.7	44.0	41.9	19.7	----	21.0
Final H_2O Content, per cent	16.9	---	22.6	25.4	32.1	33.0	37.4	39.6	28.5	27.6	19.7	21.0	16.1
Time for H_2O Addition, minutes	45	62	47	40	30	50	46	62	100	40	17	11	2
Total Mixing Time, minutes	165	1140	506	200	171	300	240	330	240	120	85	45	60
Drying Loss, per cent	1.1	4.7	4.0	4.9	--	--	--	--	--	20.7	12.5	9.5	12.3
Sintering Loss, per cent	45.9	48.2	54.6	47.9	--	--	--	--	--	52.7	51.3	47.2	43.6
Screen Analysis of Product													
plus 1 in.	1.5	--	--	14.4	--	--	--	--	2.0	--	4.4	26.3	3.5
3/4 - 1 in.	0	--	2.2	2.5	--	--	--	--	4.4	--	9.0	15.3	6.2
1/2 - 3/4 in.	1.5	--	0.8	3.2	2.6	--	--	--	9.6	--	20.3	19.7	14.1
1/4 - 1/2 in.	26.8	6.9	22.8	49.5	34.1	3.3	47.8	5.0	32.1	5.6	50.0	23.0	26.3
1/8 - 1/4 in.					50.4	43.3	20.8	24.1	41.3	20.8	5.1	9.6	32.3
3/64 - 1/8 in.					7.7	38.9	15.4	57.8	8.2	69.0	7.8	3.9	16.1
Minus 3/64 in.					5.2	14.5	16.0	13.1	2.4	4.6	3.4	2.2	1.5
6 mesh to 1/4 in.	58.2	26.8	52.1	13.3									
14 mesh to 6 mesh	11.4	59.0	18.3	12.7									
Minus 14 mesh	0.6	7.3	4.4	4.4									

Expt. No.	G-32 (d)	G-37	G-38	G-40	G-42	G-43	G-44	G-45	G-46	G-47	G-48	G-49	G-51
Dry Mix, g.	4305	1955	1955	1955	1955	1955	1955	1955	1955	1955	1955	1955	1955
Boric Acid, g.	2470	1235	1235	1235	1235	1235	1235	1235	1235	1235	1235	1235	1235
Carbon, g.	1440	720	720	720	720	720	720	720	720	720	720	720	720
Sugar, g.	395	0	0	0	0	0	0	0	0	0	0	0	0
Water Added, per cent	21.0	40.4	42.1	41.9	44.0	42.5	40.9	41.9	44.4	42.6	46.2	46.0	44.6
Final H_2O Content, per cent	16.3	22.6	--	28.5	29.7	27.0	23.1	34.2	28.9	28.2	23.4	30.2	29.2
Time for H_2O Addition, minutes	2	15	15	60	55	13	21	18	15	73	16	48	27
Total Mixing Time, minutes	60	210	225	--	90	135	90	--	50	--	90	90	90
Drying Loss, per cent	12.5	10.0	12.0	9.5	11.3	13.5	19.2	10.8	--	--	11.3	16.5	12.5
Sintering Loss, per cent	44.3	59.2	38.8	--	47.3	46.4	Combined 39.3			Combined 39.3	40.1	57.1	37.9
Screen Analysis of Product													
plus 1 in.	3.1	--	26.1	--	--	--	3.3	--	--	--	0.5	--	--
3/4 - 1 in.	6.2	1.2	3.4	--	--	--	1.8	0.8	--	--	24.2	--	--
1/2 - 3/4 in.	14.2	4.3	8.9	--	1.4	7.5	0.7	1.0	2.9	0.5	21.2	1.4	--
1/4 - 1/2 in.	26.4	9.7	27.4	4.6	5.1	48.9	3.5	5.6	12.4	2.6	18.5	7.6	11.6
1/8 - 1/4 in.	32.4	17.6	23.9	31.3	7.6	28.3	12.7	46.9	46.9	23.5	28.9	61.9	66.7
3/64 - 1/8 in.	16.2	47.8	6.9	50.2	44.1	10.7	58.8	40.8	36.6	49.6	4.2	26.7	20.1
Minus 3/64 in.	1.5	19.4	3.3	13.9	41.8	4.5	19.2	4.9	1.1	23.7	2.4	2.4	1.6

(a) A 50W Mixture

(b) Dry fines were added during latter stages of balling to restrict formation of oversize particles

(c) per cent

(d) Sigma-Blade blender used instead of rotary can

TABLE X
COMPOSITION OF SINTERED GRANULES

Expt. No.	3/4 - 1 in. diam. Granules			1/2 - 3/4 in. diam. Granules			1/4 - 1/2 in. diam. Granules		
	B Per cent	C Per cent	H Per cent	B Per cent	C Per cent	H Per cent	B Per cent	C Per cent	H Per cent
G-22	--	--	--	14.0	51.8	0.51	14.7	51.3	0.37
G-23	--	--	--	--	--	--	0.23	98.3	0.48
G-23R	--	--	--	--	--	--	0.16	99.8	0.36
G-24	--	--	--	--	--	--	1.24	97.7	0.83
G-25RR	14.0	56.2	0.46	11.5	49.5	0.69	11.2	65.4	0.83
G-26(a)	--	--	--	9.9	44.7	--	16.5	43.9	--
G-27(a)	15.4	46.4	0.59	15.3	42.2	0.79	14.9	42.6	0.83

Expt No.	1/8 - 1/4 in. diam. Granules			3/64 - 1/8 in. diam. Granules			Minus 3/64 in. Granules		
	B Per cent	C Per cent	H Per cent	B Per cent	C Per cent	H Per cent	B Per cent	C Per cent	H Per cent
G-22	15.0	43.6	0.47	12.8	57.5	0.66	10.1	63.4	0.77
G-23	0.24	96.6	0.45	0.20	97.9	0.41	0.32	97.9	0.53
G-23R	0.13	99.4	0.47	0.22	99.7	0.47	0.29	99.8	0.57
G-24	1.27	96.6	0.54	1.50	96.4	0.51	1.51	95.1	0.74
G-25RR	--	--	--	--	--	--	10.0	67.0	0.88
G-26(a)	15.6	47.1	--	13.3	53.9	--	14.2	49.6	--
G-27(a)	15.2	45.2	0.82	15.0	44.6	0.87	--	--	--

(a) Sintered at 1290°F, All others at 1000°F

TABLE XI

SUMMARY OF SINTERING DATA FOR K-150 PITCH BOUND BORIC ACID-WITCO CARBON TABLETS

Formulation	Batch No.	Sintering		Sintering Losses per cent					Hydrogen per cent		Sulfur per cent	
		Time min.	Temp. °F	Over-all	B	C	H	S	Before Sint.	After Sint.	Before Sint.	After Sint.
A-100W20K-150	25	20	1110	36.2	9.7	11.0	79	26.7	2.85	0.93	0.35	0.40
A-100W20K-150	25	40	1110	32.4	gain	0	74	gain	2.69	1.03	0.49	0.86
A-100W20K-150	25	60	1110	35.8	6.4	10.4	78	7.2	2.98	0.98	0.74	1.11
A-100W20K-150	25	120	1110	38.4	7.1	15.0	86	38	2.98	0.66	0.74	0.76
A-100W20K-150	25	180	1110	37.7	6.0	18.8	91	100	2.98	0.44	0.74	0.00
A-100W20K-150	25	20	1200	34.3	8.0	gain	84	100	3.03	0.72	0.10	0.00
A-100W20K-150	25	40	1200	33.8	7.7	gain	86	67	3.03	0.62	0.10	0.05
A-100W20K-150	25	60	1200	36.6	10.0	1.8	87	100	3.03	0.56	0.10	0.00
A-100W20K-150	25	120	1200	37.4	8.8	9.1	83	16.5	3.00	0.77	0.03	0.04
A-100W20K-150	25	180	1200	55.0	14.7	47.4	93	100	3.00	0.48	0.03	0.00
A-100W20K-150	25	20	1290	34.4	22.6	19.7	84	100	3.13	0.76	0.03	0.00
A-100W20K-150	25	40	1290	35.1	8.6	4.2	89	100	3.13	0.54	0.00	0.00
A-100W20K-150	25	60	1290	29	gain	gain	87	100	3.13	0.57	0.00	0.00

TABLE XII

SUMMARY OF SINTERING DATA FOR SUGAR-WATER BOUND BORIC ACID-CARBON TABLETS

Formulation	Batch No.	Sintering Time min.	Sintering Temp. °F	N_2 Purge	Analyses of Sintered Material Per cent B	C	H	Sintering Losses per cent Over-all (a)	B	C	H
A·100W20SuWa (80Wa)	27D	0	1110	None	8.23	38.35	3.47	--	--	--	--
A·100W20SuWa (80Wa)	27D	20	1110	None	11.88	60.63	0.34	38.2	10.9	2.4	98
A·100W20SuWa (80Wa)	27D	40	1110	None	11.97	59.37	0.26	39.2	11.7	5.9	98
A·100W20SuWa (80Wa)	27D	60	1110	None	12.33	58.15	0.18	41.0	11.5	10.6	99
A·100W20SuWa (80Wa)	27D	120	1110	None	13.17	56.57	0.21	41.6	6.4	14.3	98
A·100W20SuWa (80Wa)	27D	180	1110	None	14.35	52.53	0.15	49.0	11.8	31.3	99
A·100W20SuWa (80Wa)	27D	0	1200	None	8.28	39.01	2.99	--	--	--	--
A·100W20SuWa (80Wa)	27D	20	1200	None	11.86	60.21	0.25	39.6	13.5	6.7	97
A·100W20SuWa (80Wa)	27D	40	1200	None	12.15	59.41	0.31	42.8	16.2	12.8	96
A·100W20SuWa (80Wa)	27D	60	1200	None	12.66	57.78	0.33	44.0	14.3	16.1	96
A·100W20SuWa (80Wa)	27D	120	1200	None	14.29	51.73	0.24	52.8	18.7	36.8	97
A·100W20SuWa (80Wa)	27D	0	1290	None	8.26	38.68	3.23	--	--	--	--
A·100W20SuWa (80Wa)	27D	20	1290	None	11.94	60.67	0.26	40.0	13.2	5.9	97
A·100W20SuWa (80Wa)	27D	40	1290	None	12.25	58.44	0.32	42.0	14.0	12.3	96
A·100W20SuWa (80Wa)	27D	60	1290	None	12.31	58.62	0.19	52.0	28.4	27.2	98
A·100W20SuWa (80Wa)	27D	120	1290	None	14.02	53.11	0.17	53.6	21.2	36.2	98
A·50W15SuWa (80Wa)	28D	0	1110	None	10.88	28.5	3.03	--	--	--	--
A·50W15SuWa (80Wa)	28D	20	1110	None	16.13	47.5	0.74	51.0	27.4	18.5	87.7
A·50W15SuWa (80Wa)	28D	40	1110	None	16.54	45.17	0.25	41.8	11.4	7.75	95.5
A·50W15SuWa (80Wa)	28D	60	1110	None	16.74	44.9	0.10	41.5	12.6	10.5	98.0
A·50W15SuWa (80Wa)	28D	120	1110	None	17.28	43.48	<.1	43.1	11.8	16.0	99.0
A·50W15SuWa (80Wa)	28D	180	1110	None	18.29	38.92	0.31	46.1	12.2	27.5	95.1
A·50W15SuWa (80Wa)	28D	0	1200	None	11.01	28.43	3.36	--	--	--	--
A·50W15SuWa (80Wa)	28D	20	1200	None	16.47	45.95	0.16	41.4	13.0	5.1	97.2
A·50W15SuWa (80Wa)	28D	40	1200	None	16.74	45.75	0.12	42.1	13.0	7.0	97.9
A·50W15SuWa (80Wa)	28D	60	1200	None	17.40	43.06	<.1	42.1	10.9	14.5	98.3
A·50W15SuWa (80Wa)	28D	120	1200	None	18.43	40.17	<.1	46.4	10.4	24.2	98.3
A·50W15SuWa (80Wa)	28D	180	1200	None	19.32	36.75	<.1	49.7	11.6	34.9	98.5
A·50W15SuWa (80Wa)	28D	0	1290	None	10.55	26.8	3.54	--	--	--	--
A·50W15SuWa (80Wa)	28D	20	1290	None	16.10	43.94	0.17	43.5	17.5	12.7	97.1
A·50W15SuWa (80Wa)	28D	40	1290	None	16.89	44.54	<.1	41.0	5.5	1.9	98.3
A·50W15SuWa (80Wa)	28D	60	1290	None	16.93	44.10	.10	42.5	7.9	5.3	98.5
A·50W15SuWa (80Wa)	28D	120	1290	None	17.93	41.44	.10	45.5	7.4	13.8	99.0
A·50W15SuWa (80Wa)	28D	180	1290	None	19.14	37.23	<.10	49.0	7.6	29.0	99.0
A·100W20SuWa (80Wa)	35	0	750	None	9.04	30.85	4.00	--	--	--	--
A·100W20SuWa (80Wa)	35	20	750	None	14.26	52.72	0.41	41.5	7.96	0	94.0
A·100W20SuWa (80Wa)	35	40	750	None	14.55	52.43	0.24	42	6.6	1.4	96.5
A·100W20SuWa (80Wa)	35	60	750	None	14.20	52.83	0.26	41	7.5	-1.0 (b)	96.2
A·100W20SuWa (80Wa)	35	120	750	None	14.41	52.47	0.16	42	7.5	1.3	97.7
A·100W20SuWa (80Wa)	35	180	750	None	13.84	50.63	0.10	45	15.5	9.35	98.6
A·100W20SuWa (80Wa)	35	0	930	None	9.39	31.87	4.00	--	--	--	--
A·100W20SuWa (80Wa)	35	20	930	None	14.41	52.75	0.51	41	9.3	2.5	92.5
A·100W20SuWa (80Wa)	35	40	930	None	14.57	52.65	0.18	44	13.1	7.0	97.5
A·100W20SuWa (80Wa)	35	60	930	None	14.48	50.22	0.27	43	13.4	10.0	96.1
A·100W20SuWa (80Wa)	35	120	930	None	15.20	49.94	0.54	42	3.7	7.7	90.5
A·100W20SuWa (80Wa)	35	180	930	None	14.95	50.57	0.16	44.6	9.95	11.2	97.3
A·100W20SuWa (80Wa)	35	0	1110	None	9.48	32.38	3.75	--	--	--	--
A·100W20SuWa (80Wa)	35	20	1110	None	14.49	51.75	0.23	40.2	5.8	2.0	95.8
A·100W20SuWa (80Wa)	35	40	1110	None	14.21	52.12	0.29	41.7	12.6	6.3	95.1
A·100W20SuWa (80Wa)	35	60	1110	None	14.67	51.21	0.28	42.1	10.5	8.7	95.3
A·100W20SuWa (80Wa)	35	120	1110	None	14.94	50.53	0.45	43.6	10.5	10.5	92.5
A·100W20SuWa (80Wa)	35	180	1110	None	15.91	47.36	0.15	48.6	16 4	23.7	97.7

(a) Per cent over-all loss = $\frac{\text{wt. material in - wt. material out}}{\text{wt. material in}} \times 100$

(b) Gain in carbon

TABLE XII
SUMMARY OF SINTERING DATA FOR SUGAR-WATER BOUND BORIC ACID-CARBON TABLETS (Continued)

Formulation	Batch No.	Sintering Time min.	Sintering Temp. °F	N_2 Purge	Analyses of Sintered Material Per cent B	C	H	Sintering Losses per cent Over-all (a)	B	C	H
A·100W20SuWa (80Wa)	39	0	1110	None	9.24	29.40	3.96	--	--	--	--
A·100W20SuWa (80Wa)	39	20	1110	None	14.42	53.17	0.21	43.6	11.7	-2.0	97
A·100W20SuWa (80Wa)	39	40	1110	None	14.45	53.22	<0.1	44.0	12.5	-1.4	98
A·100W20SuWa (80Wa)	39	60	1110	None	15.14	49.75	<0.1	46.9	12.5	9.9	98
A·100W20SuWa (80Wa)	39	120	1110	None	16.70	46.76	0.0	50.5	10.4	21.1	100
A·100W20SuWa (80Wa)	39	180	1110	None	16.64	46.24	<0.1	53.6	16.5	27.0	99
A·100W20SuWa (80Wa)	39	0	1110	yes	9.27	30.41	3.83	--	--	--	--
A·100W20SuWa (80Wa)	39	20	1110	yes	13.75	54.08	0.21	40.4	11.6	-6.0	98
A·100W20SuWa (80Wa)	39	40	1110	yes	14.51	52.27	<0.1	44.0	12.2	3.7	99
A·100W20SuWa (80Wa)	39	60	1110	yes	16.01	48.58	<0.1	45.6	6.1	13.4	99
A·100W20SuWa (80Wa)	39	120	1110	yes	15.79	47.60	0.0	47.0	7.6	19.8	100
A·100W20SuWa (80Wa)	39	180	1110	yes	17.17	43.60	0.0	53.5	14.0	44.6	100
A·100W20SuWa (80Wa)	39	0	1290	yes	9.22	29.94	3.82	--	--	--	--
A·100W20SuWa (80Wa)	39	20	1290	yes	14.07	52.68	0.13	44.1	14.7	2.6	98
A·100W20SuWa (80Wa)	39	40	1290	yes	14.38	51.03	0.18	45.6	15.5	7.3	97.5
A·100W20SuWa (80Wa)	39	60	1290	yes	14.77	51.08	<0.1	46.8	14.7	9.3	99
A·100W20SuWa (80Wa)	39	120	1290	yes	15.24	50.82	<0.1	50.9	18.8	16.6	99
A·100W20SuWa (80Wa)	39	180	1290	yes	17.98	40.59	0.17	56.0	14.3	40.4	98
A·100W20SuWa (80Wa)	39	0	1290	None	9.22	29.94	3.99	--	--	--	--
A·100W20SuWa (80Wa)	39	20	1290	None	14.02	54.34	<0.1	48.3	21.5	6.1	99
A·100W20SuWa (80Wa)	39	40	1290	None	15.16	50.40	0.0	50.3	18.3	16.4	100
A·100W20SuWa (80Wa)	39	60	1290	None	15.04	50.50	0.0	45.7	11.4	8.5	100
A·100W20SuWa (80Wa)	39	120	1290	None	16.08	46.59	0.15	48.2	9.9	19.6	98
A·100W20SuWa (80Wa)	39	180	1290	None	16.95	44.10	<0.1	51.2	10.3	28.2	99
A·100W20SuWa (80Wa)	55D	0	930	yes	10.03	31.85	3.66	--	--	--	--
A·100W20SuWa (80Wa)	55D	20	930	yes	12.62	57.69	0.16	39.3	23.6	-10.2	97.3
A·100W20SuWa (80Wa)	55D	40	930	yes	12.63	57.41	0.16	39.3	23.6	-9.5	98.1
A·100W20SuWa (80Wa)	55D	60	930	yes	12.51	57.76	0.21	40.0	25.1	-8.9	98.1
A·100W20SuWa (80Wa)	55D	120	930	yes	12.71	57.13	0.16	41.3	25.5	-5.4	98.1
A·100W20SuWa (80Wa)	55D	180	930	yes	12.62	56.67	0.20	47.8	34.4	7.1	97.3
A·100W20SuWa (80Wa)	55D	0	1110	yes	10.03	31.85	3.66	--	--	--	--
A·100W20SuWa (80Wa)	55D	20	1110	yes	12.48	57.37	<0.1	39.3	24.4	-9.3	98.4
A·100W20SuWa (80Wa)	55D	40	1110	yes	12.66	57.38	0.0	44.3	29.7	0	100.0
A·100W20SuWa (80Wa)	55D	60	1110	yes	14.20	53.41	0.0	47.1	25.2	11.4	100.0
A·100W20SuWa (80Wa)	55D	120	1110	yes	15.23	49.61	<0.1	52.0	27.0	25.2	98.7
A·100W20SuWa (80Wa)	55D	180	1110	yes	15.86	48.29	0.0	53.7	26.8	29.9	100.0
A·100W20SuWa (80Wa)	55D	0	1200	yes	10.03	31.85	3.66	--	--	--	--
A·100W20SuWa (80Wa)	55D	20	1200	yes	12.50	60.53	0.0	41.4	27.0	-11.2	100.0
A·100W20SuWa (80Wa)	55D	40	1200	yes	14.21	53.43	0.0	42.0	17.7	3.1	100.0
A·100W20SuWa (80Wa)	55D	60	1200	yes	14.83	51.55	0.15	44.3	17.5	9.6	97.0
A·100W20SuWa (80Wa)	55D	120	1200	yes	16.94	44.37	0.11	50.9	17.0	31.4	98.5
A·100W20SuWa (80Wa)	55D	180	1200	yes	17.10	44.29	0.0	52.6	19.1	34	100.0
A·100W20SuWa (80Wa)	55D	0	1290	yes	10.03	31.85	3.66	--	--	--	--
A·100W20SuWa (80Wa)	55D	20	1290	yes	13.81	54.63	<0.1	40.9	18.0	-1.4	98.4
A·100W20SuWa (80Wa)	55D	40	1290	yes	13.72	54.71	0.13	41.4	19.8	-1.7	97.9
A·100W20SuWa (80Wa)	55D	60	1290	yes	13.61	54.68	0.17	47.4	28.6	9.4	97.6
A·100W20SuWa (80Wa)	55D	120	1290	yes	14.03	49.75	0.14	52.5	33.5	25.8	99.0
A·100W20SuWa (80Wa)	55D	180	1290	yes	16.35	46.88	<0.1	55.3	38.8	32.8	98.8
A·100W20SuWa (60Wa)	98	0	570	yes	9.31	31.44	3.43	--	--	--	--
A·100W20SuWa (60Wa)	98	20	570	yes	12.61	52.39	1.25	33.5	9.7	-10.7	76
A·100W20SuWa (60Wa)	98	40	570	yes	13.22	54.61	1.30	38.0	11.8	-7.9	77
A·100W20SuWa (60Wa)	98	60	570	yes	13.37	55.48	1.24	38.0	10.8	-9.5	77
A·100W20SuWa (60Wa)	98	120	570	yes	13.41	52.63	0.77	38.2	10.8	-3.5	86
A·100W20SuWa (60Wa)	98	180	570	yes	12.71	52.62	1.63	38.5	16.1	-2.9	71
A·100W20SuWa (60Wa)	98	0	750	yes	9.31	31.44	3.43	--	--	--	--
A·100W20SuWa (60Wa)	98	20	750	yes	14.36	55.09	0.52	39.5	6.5	-6.2	91
A·100W20SuWa (60Wa)	98	40	750	yes	14.22	54.90	0.44	39.0	6.7	-6.5	92
A·100W20SuWa (60Wa)	98	60	750	yes	13.91	55.54	0.44	39.0	8.6	-7.9	92
A·100W20SuWa (60Wa)	98	120	750	yes	14.15	54.83	0.35	40.2	9.1	-4.4	94
A·100W20SuWa (60Wa)	98	180	750	yes	14.20	54.44	0.41	40.4	9.1	-3.0	93
A·100W20SuWa (60Wa)	98	0	930	yes	9.31	31.44	3.43	--	--	--	--
A·100W20SuWa (60Wa)	98	20	930	yes	14.27	54.37	0.35	40.0	8.1	-2.9	94
A·100W20SuWa (60Wa)	98	40	930	yes	13.72	54.24	0.27	40.7	12.6	-2.1	96
A·100W20SuWa (60Wa)	98	60	930	yes	13.80	54.54	0.27	41.3	12.9	-1.9	96
A·100W20SuWa (60Wa)	98	120	930	yes	14.52	51.38	0.15	43.7	12.1	7.7	97
A·100W20SuWa (60Wa)	98	180	930	yes	15.11	50.14	0.34	44.3	9.1	10.8	94

(a) Per cent over-all loss = $\frac{\text{wt. material in - wt. material out}}{\text{wt. material in}} \times 100$

(b) Gain in carbon

TABLE XIII

SUMMARY OF SINTERING DATA FOR MOLASSES-WATER BOUND BORIC ACID-CARBON TABLETS

Formulation	Batch No.	Sintering Time min.	Sintering Temp. °F	N_2 Purge	Analysis of Sintered Material (a) Per cent B	Per cent C	Per cent H	Sintering Losses, per ce Over-all (b)	B	C
A·100W20MWA (60Wa)	74	0	570	yes	9.59	34.10	3.54	--	--	--
A·100W20MWA (60Wa)	74	20	570	yes	13.04	51.12	0.64	32.5	8.2	-.11
A·100W20MWA (60Wa)	74	40	570	yes	13.54	53.76	0.50	36.5	10.5	-.04
A·100W20MWA (60Wa)	74	60	570	yes	13.30	53.28	0.60	36.8	12.4	1.3
A·100W20MWA (60Wa)	74	120	570	yes	13.59	53.60	0.38	37.1	10.7	1.1
A·100W20MWA (60Wa)	74	180	570	yes	13.50	53.81	0.27	36.2	10.2	-.57
A·100W20MWA (60Wa)	74	0	750	yes	9.59	34.10	3.54	--	--	--
A·100W20MWA (60Wa)	74	20	750	yes	13.46	54.07	0.26	37.9	12.7	1.47
A·100W20MWA (60Wa)	74	40	750	yes	13.38	54.16	0.34	38.4	14.1	2.2
A·100W20MWA (60Wa)	74	60	750	yes	13.13	54.19	0.30	38.4	15.7	2.1
A·100W20MWA (60Wa)	74	120	750	yes	13.42	53.90	0.30	38.7	14.1	3.1
A·100W20MWA (60Wa)	74	180	750	yes	13.43	53.32	0.21	38.7	14.1	4.1
A·100W20MWA (60Wa)	74	0	930	yes	9.59	34.10	3.54	--	--	--
A·100W20MWA (60Wa)	74	20	930	yes	13.63	52.51	0.24	40.0	14.6	7.6
A·100W20MWA (60Wa)	74	40	930	yes	13.05	53.60	0.19	40.3	18.8	6.1
A·100W20MWA (60Wa)	74	60	930	yes	14.00	51.70	0.23	40.3	12.7	9.4
A·100W20MWA (60Wa)	74	120	930	yes	14.25	51.20	< 0.10	41.6	13.2	12.3
A·100W20MWA (60Wa)	74	180	930	yes	14.80	49.00	< 0.10	44.3	14.1	19.9
A·100W20MWA (60Wa)	74	0	1110	yes	9.59	34.10	3.54	--	--	--
A·100W20MWA (60Wa)	74	20	1110	yes	13.90	52.05	0.17	40.3	13.5	8.8
A·100W20MWA (60Wa)	74	40	1110	yes	14.55	51.10	0.16	42.7	13.0	14.1
A·100W20MWA (60Wa)	74	60	1110	yes	14.95	51.45	<0.10	44.3	13.2	15.9
A·100W20MWA (60Wa)	74	120	1110	yes	15.95	47.20	0.16	48.5	14.3	28.8
A·100W20MWA (60Wa)	74	180	1110	yes	17.35	41.70	0.13	46.7	3.5	34.8

(a) Residual ash contents at 570, 750, 930, and 1110°F were 2.08, 2.39, 2.15 and 2.70 per cent, respectively

(b) Per cent Over-all Loss = $\frac{\text{wt. material in - wt. material out}}{\text{wt. material in}} \times 100$

TABLE XIV
SUMMARY OF SINTERING DATA FOR STARCH-WATER BOUND BORIC ACID - CARBON TABLETS

Formulation	Batch No.	Sintering Time min.	Sintering Temp. °F	N_2 Purge	Analyses of Sintered Material Per cent B	C	H	Sintering Losses, per cent Over-all	B	C	H
A-100W25SWA (80Wa)	43	0	1110	None	9.09	35.77	3.53	--	--	--	--
A-100W25SWA (80Wa)	43	180	1110	None	17.1	44.15	<0.1	53.4	12.4	42.5	99
A-100W25SWA (80Wa)	43	0	1200	None	9.09	35.77	3.53	--	--	--	--
A-100W25SWA (80Wa)	43	20	1200	None	13.94	52.74	0.31	43.8	13.7	17.0	95
A-100W25SWA (80Wa)	43	40	1200	None	14.78	51.09	0.19	46.9	13.1	24.1	98
A-100W25SWA (80Wa)	43	60	1200	None	15.76	47.27	0.20	50.0	13.2	33.8	98
A-100W25SWA (80Wa)	43	120	1200	None	17.16	43.41	0.17	54.1	13.2	42.9	98
A-100W25SWA (80Wa)	43	180	1200	None	17.83	41.68	0.16	55.9	13.2	50.5	98
A-100W25SWA (80Wa)	43	0	1290	None	9.09	35.77	3.53	--	--	--	--
A-100W25SWA (80Wa)	43	20	1290	None	14.32	52.68	0.44	44.5	12.6	18.0	93
A-100W25SWA (80Wa)	43	40	1290	None	14.89	50.67	0.27	46.2	12.0	23.6	96
A-100W25SWA (80Wa)	43	60	1290	None	16.24	45.83	0.22	51.8	13.8	38.2	98
A-100W25SWA (80Wa)	43	120	1290	None	17.82	41.75	0.13	55.1	11.5	47.6	98
A-100W25SWA (80Wa)	43	180	1290	None	18.61	38.61	0.14	57.6	13.2	54.5	98
A-100W25SWA (80Wa)	54	0	932	Yes	9.53	28.97	3.70	--	--	--	--
A-100W25SWA (80Wa)	54	20	932	Yes	13.95	54.27	0.21	42.9	16.4	-7.0	97
A-100W25SWA (80Wa)	54	40	932	Yes	14.05	55.08	0.27	45.1	19.2	-4.3	96
A-100W25SWA (80Wa)	54	60	932	Yes	13.93	52.93	0.29	45.1	19.8	0	96
A-100W25SWA (80Wa)	54	120	932	Yes	14.41	53.25	0.22	46.3	18.7	1.3	97
A-100W25SWA (80Wa)	54	180	932	Yes	14.77	51.20	0.18	47.6	18.6	7.2	97+
A-100W25SWA (80Wa)	54	0	1110	Yes	9.53	28.97	3.70	--	--	--	--
A-100W25SWA (80Wa)	54	20	1110	Yes	14.07	53.50	0.12	45.4	19.5	-7.9	98
A-100W25SWA (80Wa)	54	40	1110	Yes	14.06	54.36	0.22	46.0	20.4	-1.2	97
A-100W25SWA (80Wa)	54	60	1110	Yes	15.52	50.43	0.19	50.3	18.9	13.5	97+
A-100W25SWA (80Wa)	54	120	1110	Yes	16.89	44.17	0.20	54.6	19.4	30.9	97+
A-100W25SWA (80Wa)	54	180	1110	Yes	16.81	44.57	0.12	59.1	27.9	37.1	99
A-100W25SWA (80Wa)	54	0	1200	Yes	9.53	28.97	3.70	--	--	--	--
A-100W25SWA (80Wa)	54	20	1200	Yes	13.29	55.16	0.19	46.3	25.2	-2.3	97
A-100W25SWA (80Wa)	54	40	1200	Yes	13.77	53.87	0.21	47.7	24.4	2.8	97
A-100W25SWA (80Wa)	54	60	1200	Yes	15.34	49.22	0.29	50.3	19.3	15.5	96
A-100W25SWA (80Wa)	54	120	1200	Yes	16.15	46.37	0.16	52.6	19.6	24.2	98
A-100W25SWA (80Wa)	54	180	1200	Yes	17.87	40.89	0.12	57.4	20.2	39.9	99
A-100W25SWA (80Wa)	54	0	1290	Yes	9.53	28.97	3.70	--	--	--	--
A-100W25SWA (80Wa)	54	20	1290	Yes	14.23	52.78	0.16	45.7	18.9	1.0	98
A-100W25SWA (80Wa)	54	40	1290	Yes	15.17	45.71	0.22	49.4	19.6	20.2	97
A-100W25SWA (80Wa)	54	60	1290	Yes	16.38	45.90	0.19	52.9	19.0	25.3	98
A-100W25SWA (80Wa)	54	120	1290	Yes	18.40	39.48	0.17	57.4	17.8	41.9	98
A-100W25SWA (80Wa)	54	180	1290	Yes	20.75	31.40	0.0	62.9	19.2	59.8	100
A-100W25SWA (80Wa)	56	0	300	Yes	10.67	37.83	3.92	--	--	--	--
A-100W25SWA (80Wa)	56	20	300	Yes	13.30	51.00	0.43	36.5	20.8	14.4	93.0
A-100W25SWA (80Wa)	56	40	300	Yes	13.43	51.24	0.57	38.3	22.91	16.33	91.0
A-100W25SWA (80Wa)	56	60	300	Yes	13.52	53.25	0.42	38.8	22.40	13.80	93.4
A-100W25SWA (80Wa)	56	120	300	Yes	13.41	51.52	0.44	37.5	21.44	14.90	93.0
A-100W25SWA (80Wa)	56	180	300	Yes	12.37	49.29	1.17	32.0	21.16	11.40	79.7
A-100W25SWA (80Wa)	56	0	390	Yes	10.67	37.83	3.92	--	--	--	--
A-100W25SWA (80Wa)	56	20	390	Yes	16.81	45.20	2.86	13.7	-3.6	-3.1	37.0
A-100W25SWA (80Wa)	56	40	390	Yes	17.91	41.39	2.18	25.7	-2.5	18.72	58.7
A-100W25SWA (80Wa)	56	60	390	Yes	18.65	38.22	1.55	28.3	-2.53	27.54	71.7
A-100W25SWA (80Wa)	56	120	390	Yes	18.10	39.61	1.07	31.4	-1.63	28.20	81.3
A-100W25SWA (80Wa)	56	180	390	Yes	15.61	44.95	0.75	35.4	5.5	18.13	87.6
A-100W25SWA (80Wa)	56	0	570	Yes	10.67	37.83	3.92	--	--	--	--
A-100W25SWA (80Wa)	56	20	570	Yes	13.13	41.63	1.51	3.01	14.22	23.30	73.2
A-100W25SWA (80Wa)	56	40	570	Yes	13.50	51.11	0.55	37.1	20.50	15.10	91.2
A-100W25SWA (80Wa)	56	60	570	Yes	13.41	52.70	0.41	38.6	22.90	14.40	93.6
A-100W25SWA (80Wa)	56	120	570	Yes	13.35	53.88	0.24	38.9	23.40	12.90	96.3
A-100W25SWA (80Wa)	56	180	570	Yes	13.44	54.88	0.34	40.0	24.42	13.00	94.8
A-100W25SWA (80Wa)	56	0	750	Yes	10.67	37.83	3.92	--	--	--	--
A-100W25SWA (80Wa)	56	20	750	Yes	13.42	55.31	0.19	37.3	32.45	21.50	96.9
A-100W25SWA (80Wa)	56	40	750	Yes	13.49	55.32	0.24	39.0	33.90	23.54	96.2
A-100W25SWA (80Wa)	56	60	750	Yes	13.51	55.17	0.23	38.7	22.34	10.60	96.4
A-100W25SWA (80Wa)	56	120	750	Yes	13.56	54.95	0.20	39.7	23.33	12.40	96.9
A-100W25SWA (80Wa)	56	180	750	Yes	13.55	55.17	0.19	40.1	24.01	12.03	97.1
A-100W25SWA (80Wa)	56D	0	300	Yes	10.13	34.87	3.13	--	--	--	--
A-100W25SWA (80Wa)	56D	20	300	Yes	13.42	51.22	0.85	33.5	11.9	2.4	81.9
A-100W25SWA (80Wa)	56D	40	300	Yes	13.16	52.90	0.45	33.8	12.4	-.3	90.5
A-100W25SWA (80Wa)	56D	60	300	Yes	13.00	50.77	0.58	32.9	13.9	2.3	87.6
A-100W25SWA (80Wa)	56D	120	300	Yes	13.06	49.52	0.72	52	38.2	31.8	89.0
A-100W25SWA (80Wa)	56D	180	300	Yes	12.43	49.59	1.22	48	36.2	26.0	79.7
A-100W25SWA (80Wa)	56D	0	390	Yes	10.13	34.87	3.13	--	--	--	--
A-100W25SWA (80Wa)	56D	20	390	Yes	11.15	43.54	2.29	15.4	6.9	-5.6	38.1
A-100W25SWA (80Wa)	56D	40	390	Yes	12.39	47.63	1.34	23.4	6.3	-4.7	67.2
A-100W25SWA (80Wa)	56D	60	390	Yes	12.63	51.37	0.93	30.8	3.7	-2.0	69.6
A-100W25SWA (80Wa)	56D	120	390	Yes	13.10	50.00	0.76	31.4	9.9	1.6	83.4
A-100W25SWA (80Wa)	56D	180	390	Yes	13.20	49.90	0.71	30.8	9.8	0.9	84.3
A-100W25SWA (80Wa)	56D	0	570	Yes	10.13	34.87	3.13	--	--	--	--
A-100W25SWA (80Wa)	56D	20	570	Yes	12.91	49.74	0.79	30.5	11.4	0.8	82.4
A-100W25SWA (80Wa)	56D	40	570	Yes	13.56	53.06	0.58	34.5	12.3	0.2	87.8
A-100W25SWA (80Wa)	56D	60	570	Yes	13.54	56.56	0.46	34.8	12.8	-5.8	90.4
A-100W25SWA (80Wa)	56D	120	570	Yes	13.05	57.64	0.31	37.8	20.0	-2.7	93.8
A-100W25SWA (80Wa)	56D	180	570	Yes	13.95	53.10	0.16	35.4	11.0	-1.6	96.7
A-100W25SWA (80Wa)	56D	0	750	Yes	10.13	34.87	3.13	--	--	--	--
A-100W25SWA (80Wa)	56D	20	750	Yes	13.07	57.92	0.22	35.7	17.0	-6.8	95.5
A-100W25SWA (80Wa)	56D	40	750	Yes	13.53	55.70	0.22	36.0	14.5	-2.2	95.5
A-100W25SWA (80Wa)	56D	60	750	Yes	13.8	53.74	0.30	36.3	12.8	1.9	93.9
A-100W25SWA (80Wa)	56D	120	750	Yes	15.4	52.59	0.19	36.0	2.2	3.4	96.1
A-100W25SWA (80Wa)	56D	180	750	Yes	14.85	49.65	0.11	36.0	6.2	8.9	97.3
A-100W25SWA (80Wa)	45	0	1290	None	10.08	32.31	2.80	--	--	--	--
A-100W25SWA (80Wa)	45	40	1290	None	14.17	53.14	0.30	42.8	19.5	5.7	93.8
A-100W25SWA (80Wa)	45	40	1290	None	14.17	52.90	0.21	43.5	20.1	7.9	96.7
A-100W25SWA (80Wa)	45	40	1290	None	13.88	53.68	0.17	42.8	21.1	4.8	96.5
A-100W25SWA (80Wa)	45	40	1290	None	13.96	54.57	0.24	43.0	21.1	3.6	95.1
A-100W25SWA (80Wa)	45	40	1290	None	14.21	53.02	0.20	43.3	19.9	5.0	96.0
A-100W25SWA (80Wa)	60	0	1290	None	9.03	34.02	3.84	--	--	--	--
A-100W25SWA (80Wa)	60	40	1290	None	17.32	43.5	0.20	46.0	-3.5	31.0	97.2
A-100W25SWA (80Wa)	60	40	1290	None	17.27	43.9	<0.1	45.0	4.9	29.0	>98.6
A-100W25SWA (80Wa)	60	40	1290	None	17.80	43.75	0.0	45.8	-7.0	30.2	100.0
A-100W25SWA (80Wa)	60	40	1290	None	16.98	44.63	<0.1	46.0	-1.5	29.2	>98.6
A-100W25SWA (80Wa)	60	40	1290	None	17.10	44.90	<0.1	45.3	-3.7	27.8	>98.6

(a) Per cent over all loss = $\frac{\text{wt. material in} - \text{wt. material out}}{\text{wt. material in}} \times 100$

TABLE XV
RELATIVE SINTERING LOSSES OF K-150 PITCH MOLLASSES STARCH AND SUGAR BOUND TABLETS

Formulation	Batch No.	Sintering Time min.	Sintering Temp. °F	N_2 Purge	Residual Hydrogen Per cent	Sintering Losses Per cent Over all	B	C	H
A·100W20SuWA (80WA)	55D	60	930	Yes	0.20	40	23	-9	98
A·100W20SuWA (80WA)	76	60	930	No	0.26	41	15.7	7	99
A·100W20MWA (60WA)	74	60	930	Yes	0.16	40	14.5	8	97
A·100W25SWA (80WA)	54	60	930	Yes	0.26	45	19	-4	96
A·100W·20K150	25	60	1110	No	0.97	36	9	3	78

TABLE XVI
SUMMARY OF SINTERING DATA FOR STARCH-WATER BOUND BORIC ACID-CARBON BRIQUETS

Formulation	Batch No.	Sintering Time Min.	Sintering Temp. °F	N_2 Purge	Analysis of Sintered Material Per cent B	C	H	Sintering Losses Per cent Overall	B	C	H
A-100W25, 4SWA(88WA) Bldg. 47 Briquets	46	0	930	None	9.27	29.51	3.96	--	--	--	--
A-100W25, 4SWA(88WA) Bldg. 47 Briquets	46	20	930	None	15.06	50.69	0.25	46.5	13.05	8.10	96.6
A-100W25, 4SWA(88WA) Bldg. 47 Briquets	46	40	930	None	14.96	49.76	0.23	46.9	13.05	8.10	96.9
A-100W25, 4SWA(88WA) Bldg. 47 Briquets	46	60	930	None	15.20	49.72	0.21	47.4	13.80	11.10	97.2
A-100W25, 4SWA(88WA) Bldg. 47 Briquets	46	120	930	None	14.97	50.05	<0.10	48.0	16.00	11.10	98.7
A-100W25, 4SWA(88WA) Bldg. 47 Briquets	46	180	930	None	15.24	50.27	0.12	48.3	12.00	15.05	98.4
A-100W25, 4SWA(88WA) Bldg. 47 Briquets	46	0	930	Yes	9.27	29.51	3.96	--	--	--	--
A-100W25, 4SWA(88WA) Bldg. 47 Briquets	46	20	930	Yes	15.13	50.71	0.10	46.5	12.70	8.10	98.6
A-100W25, 4SWA(88WA) Bldg. 47 Briquets	46	40	930	Yes	15.05	49.86	0.13	47.6	14.80	11.50	98.3
A-100W25, 4SWA(88WA) Bldg. 47 Briquets	46	60	930	Yes	14.86	51.19	0.11	48.0	16.60	9.70	98.5
A-100W25, 4SWA(88WA) Bldg. 47 Briquets	46	120	930	Yes	15.10	49.84	0.15	48.7	16.44	13.34	98.0
A-100W25, 4SWA(88WA) Bldg. 47 Briquets	46	180	930	Yes	15.00	49.36	0.20	49.8	18.80	16.10	97.5
A-100W25, 4SWA(88WA) Bldg. 47 Briquets	46	0	1110	Yes	9.27	29.51	3.96	--	--	--	--
A-100W25, 4SWA(88WA) Bldg. 47 Briquets	46	20	1110	Yes	14.96	49.71	0.11	48.5	17.10	13.00	98.6
A-100W25, 4SWA(88WA) Bldg. 47 Briquets	46	40	1110	Yes	14.86	47.54	0.17	49.1	18.3	29.6	97.9
A-100W25, 4SWA(88WA) Bldg. 47 Briquets	46	60	1110	Yes	16.24	46.41	0.16	52.4	16.60	25.10	98.0
A-100W25, 4SWA(88WA) Bldg. 47 Briquets	46	120	1110	Yes	16.84	44.17	<0.10	53.9	16.70	31.47	98.9
A-100W25, 4SWA(88WA) Bldg. 47 Briquets	46	180	1110	Yes	17.67	42.33	0.11	56.5	16.80	37.48	98.8
A-100W25, 4SWA(88WA) Bldg. 47 Briquets	46	0	1200	Yes	9.27	29.51	3.96	--	--	--	--
A-100W25, 4SWA(88WA) Bldg. 47 Briquets	46	20	1200	Yes	15.23	49.03	0.32	46.9	12.70	11.80	95.7
A-100W25, 4SWA(88WA) Bldg. 47 Briquets	46	40	1200	Yes	15.92	46.40	0.14	52.2	17.94	24.80	98.3
A-100W25, 4SWA(88WA) Bldg. 47 Briquets	46	60	1200	Yes	16.81	45.20	0.20	53.5	15.80	28.84	97.6
A-100W25, 4SWA(88WA) Bldg. 47 Briquets	46	120	1200	Yes	17.91	41.39	0.00	56.8	16.60	39.43	100.0
A-100W25, 4SWA(88WA) Bldg. 47 Briquets	46	180	1200	Yes	18.10	39.61	<0.1	58.5	18.92	44.30	99.0
A-100W25, 4SWA(88WA) Bldg. 47 Briquets	46	0	1290	Yes	9.27	29.51	3.96	--	--	--	--
A-100W25, 4SWA(88WA) Bldg. 47 Briquets	46	20	1290	Yes	15.61	47.95	<0.1	52.8	20.50	23.20	98.8
A-100W25, 4SWA(88WA) Bldg. 47 Briquets	46	40	1290	Yes	13.11	41.63	1.46	53.9	34.71	34.92	83.0
A-100W25, 4SWA(88WA) Bldg. 47 Briquets	46	60	1290	Yes	17.10	43.90	0.22	55.4	17.60	33.60	97.5
A-100W25, 4SWA(88WA) Bldg. 47 Briquets	46	120	1290	Yes	17.45	39.05	0.39	59.5	33.12	53.11	96.1
A-100W25, 4SWA(88WA) Bldg. 47 Briquets	46	180	1290	Yes	18.00	38.15	0.15	58.3	22.61	48.50	98.4
A-100W25, 4SWA(88WA) Bldg. 47 Briquets	46	0	1290	None	9.27	29.51	3.96	--	--	--	--
A-100W25, 4SWA(88WA) Bldg. 47 Briquets	46	20	1290	None	15.15	49.25	0.12	45.6	11.10	9.22	98.4
A-100W25, 4SWA(88WA) Bldg. 47 Briquets	46	40	1290	None	15.50	48.60	0.14	48.4	13.70	15.02	98.2
A-100W25, 4SWA(88WA) Bldg. 47 Briquets	46	60	1290	None	16.30	45.50	0.0	50.4	12.80	23.53	99.0
A-100W25, 4SWA(88WA) Bldg. 47 Briquets	46	120	1290	None	18.30	38.65	0.16	55.2	28.80	50.80	98.5
A-100W25, 4SWA(88WA) Bldg. 47 Briquets	46	180	1290	None	20.24	36.29	0.0	63.3	32.70	62.13	99.0

TABLE XVII
SUMMARY OF SINTERING DATA FOR SUGAR-WATER BOUND BORIC ACID-CARBON BRIQUETS (a)

Formulation	Batch No.	Sintering Time min.	Sintering Temp. °F	Purge	Analysis of Sintered Briquets Per cent B	Analysis of Sintered Briquets Per cent C	Analysis of Sintered Briquets Per cent H	Sintering Losses Per cent Overall	Sintering Losses Per cent B	Sintering Losses Per cent C	Sintering Losses Per cent H
A-100W-20SuWa(60Wa)	99	20	570	Yes	12.66	50.01	1.30	31.8	3.9	-12.1	76.0
A-100W-20SuWa(60Wa)	99	40	570	Yes	12.71	55.71	0.74	38.0	12.5	-13.6	78.0
A-100W-20SuWa(60Wa)	99	60	570	Yes	13.11	57.06	0.36	36.0	7.8	-18.7	93.7
A-100W-20SuWa(60Wa)	99	120	570	Yes	13.05	56.53	0.45	39.1	11.4	-13.4	92.5
A-100W-20SuWa(60Wa)	99	180	570	Yes	13.19	55.79	0.46	38.3	9.7	-13.3	87.0
A-100W-20SuWa(60Wa)	99	20	750	Yes	12.51	57.54	0.48	40.8	17.5	-12.1	90.3
A-100W-20SuWa(60Wa)	99	40	750	Yes	13.04	56.55	0.29	41.3	15.0	- 9.2	95.1
A-100W-20SuWa(60Wa)	99	60	750	Yes	13.23	56.64	0.33	40.8	12.8	-10.3	94.6
A-100W-20SuWa(60Wa)	99	120	750	Yes	13.24	56.10	0.41	43.8	17.2	- 3.7	93.8
A-100W-20SuWa(60Wa)	99	180	750	Yes	13.50	55.29	0.33	41.6	12.2	- 6.3	95.1
A-100W-20SuWa(60Wa)	99	20	930	Yes	13.14	56.83	0.37	42.0	16.1	- 8.3	93.8
A-100W-20SuWa(60Wa)	99	40	930	Yes	13.48	55.71	0.25	41.8	12.8	- 6.6	96.0
A-100W-20SuWa(60Wa)	99	60	930	Yes	14.12	54.61	0.12	43.6	11.5	- 1.6	98.0
A-100W-20SuWa(60Wa)	99	120	930	Yes	14.20	53.78	0.10	43.6	10.6	- 0.3	98.0
A-100W-20SuWa(60Wa)	99	180	930	Yes	14.60	52.62	0.27	46.6	13.0	7.7	96.0

(a) Analysis of green briquets: Per cent B = 9.00
Per cent C = 30.44
Per cent H = 3.64

TABLE XVIII

SINTERING DATA FOR BORIC ACID-CARBON GRANULES

Expt. No.	Description of Material	Size Fraction	Sintering Conditions	Weight Analysis of Product				Weight Out	B Loss	C Loss
				N Per cent	B Per cent	C Per cent	H Per cent	Per cent	Per cent	Per cen
RG-138	Powdered BA+C+H_2O spray	Minus 6 mesh	Before	--	10.2	31.7	329	--	--	--
			1000°F 1 hr.	400	15.1	48.2	0.67	187	30.9	29.0
			1000°F 1 hr.	400	15.3	45.7	0.79	176	34.1	36.6
		6 mesh to 1/4 in.	Before	--	9.47	30.9	3.74	--	--	--
			1000°F 1 hr.	400	14.3	43.5	0.97	186	29.6	34.6
			1000°F 1 hr.	400	14.8	47.4	0.60	184	28.1	29.4
		1/4 in. to 3/8 in.	Before	--	10.7	36.1	3.26	--	--	--
			1000°F 1 hr.	400	14.1	45.4	0.95	192	36.7	39.5
		3/8 in. to 1 2 in.	Before	--	11.3	36.8	2.77	--	--	--
			1000°F 1 hr.	400	15.0	47.7	0.45	225	25.4	27.0
RG-139	Powdered BA+C + 5 Per cent BA spray	6 mesh to 1/4 in.	Before	--	12.0	37.7	2.41	--	--	--
			1000°F 1 hr.	400	14.5	49.1	0.61	197	40.3	35.8
			1000°F 1 hr.	400	16.5	43.9	0.34	213	35.2	38.0
		1/4 in. to 3/8 in.	Before	--	9.01	30.1	3.98	--	--	--
			1000°F 1 hr.	400	16.0	45.7	0.38	197	12.5	25.2
			1000°F 1 hr.	400	11.8	47.3	0.43	199	34.7	21.8
		3/8 in. to 1/2 in.	Before	--	8.34	32.4	4.10	--	--	--
			1000°F 1 hr.	400	13.5	53.3	0.40	201	18.6	17.3
			1000°F 1 hr.	400	14.9	49.0	0.58	204	8.7	22.8
		Plus 1/2 in.	Before	--	8.48	29.7	3.95	--	--	--
			1000°F 1 hr.	400	14.9	49.4	0.39	208	8.5	13.5
			1000°F 1 hr.	400	14.7	49.4	0.42	202	12.4	16.0
RG-140	Granualar BA+C +H_2O spray	6 mesh to 1/4 in.	Before	--	8.94	29.1	4.05	--	--	--
			1000°F 1 hr.	400	15.2	46.8	0.64	192	18.4	22.4
			1000°F 1 hr.	400	15.0	47.9	0.58	189	20.6	22.2
		1/4 in. to 3/8 in.	Before	--	9.12	29.6	3.77	--	--	--
			1000°F 1 hr.	400	14.5	49.0	0.86	191	24.0	20.9
			1000°F 1 hr.	400	15.2	49.2	0.57	199	17.3	17.3
		3/8 in. to 1/2 in.	Before	--	10.4	3.14	4.05	--	--	--
			1000°F 1 hr.	400	14.9	49.8	0.36	205	27.5	17.1
			1000°F 1 hr.	400	15.3	49.0	0.58	202	26.6	21.1

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TABLE XIX
SINTERING DATA FOR SODIUM TETRABORATE WITCO CARBON TABLETS; STOICHIOMETRIC BORON-CARBON RATIO (a)

Sintering Conditions	Final Analysis			
	Per cent B	Per cent C	Per cent H	B/C
1 hr. 194°F plus 1 hr. 500°F	14.5	31.0	0.56	0.47
2 hr. 194°F plus 2 hr. 500°F	13.7	29.5	0.50	0.47
1 hr. 194°F plus 1 hr. 750°F	14.7	30.2	0.23	0.49
2 hr. 194°F plus 2 hr. 750°F	14.9	28.7	0.33	0.52
1 hr. 194°F plus 1 hr. 900°F	18.0	15.1	0.13	1.19
2 hr. 194°F plus 2 hr. 900°F	19.3	9.25	0.09	2.09

(a) Theoretical B/C assuming no sintering losses = 0.515

TABLE XX
SUMMARY OF SINTERING DATA FOR
SUGAR-WATER BOUND CARBON TABLETS

Formulation	Batch No.	Sintering Time Min.	Sintering Temp. °F	Purge	Residual Hydrogen Per cent H	Over-all Sintering Loss Per cent
67W.33SuWa(70Wa)	38	20	750	No	0.42	34.8
		40	750	No	0.35	31.2
		60	750	No	0.22	18.4
		120	750	No	0.26	32.8
		180	750	No	0.46	33.5
		20	930	No	0.39	32.5
		40	930	No	0.24	33.9
		60	930	No	0.41	38.0
		120	930	No	0.45	45.6
		180	930	No	0.26	68.0
		20	1110	No	0.32	28.4
		40	1110	No	0.45	38.4
		60	1110	No	0.14	22.0
		120	1110	No	0.43	25.8
		180	1110	No	0.43	82.5
		20	1200	No	0.29	39.5
		40	1200	No	0.37	42.5
		60	1200	No	0.30	51.0
		120	1200	No	0.34	43.2
		180	1200	No	0.23	50.8
		20	1290	No	0.39	35.2
		40	1290	No	0.29	34.4
		60	1290	No	0.28	38.3
		120	1290	No	0.28	47.9
		180	1290	No	0.31	39.6

TABLE XXI
EFFECT OF PREDRYING ON SINTERING LOSSES OF TABLETTED FEED

Formulation	Batch No.	Predrying Time Min.	Predrying Temp. °F	Drying Loss Per cent	Analyses After Sintering (a) Per cent B	C	H	Sintering Losses per cent Overall	B	C
A- 100W20SuWa(80Wa)	76(b)	Not Predried			14.54	52.70	0.37	41.2	15.6	5.674
		Not Predried			14.38	52.59	0.23	41.2	15.6	5.870
		Not Predried			14.39	51.75	0.18	41.2	15.9	5.370
		60	176	8.0	14.20	52.65	0.26	40.0	15.9	3.840
		60 Predried		10.0	14.74	51.37	0.31	40.4	14.3	3.890
		60 Predried		12.4	14.43	52.97	0.26	39.6	13.9	2.620
		60 Predried	212	10.8	14.21	51.94	0.34	40.0	15.8	5.470
		60 Predried		8.0	14.21	50.65	0.42	40.0	15.8	7.480
		60 Predried		10.4	14.24	51.71	0.20	40.3	16.2	6.190
		60 Predried	248	17.2	14.42	51.46	0.19	39.2	13.4	4.760
		60 Predried		16.0	14.67	50.79	0.23	39.6	12.5	6.610
		60 Predried		16.8	14.60	51.15	0.18	38.8	11.8	4.712
	97(c)	Not Predried			14.56	51.02	0.37	42.3	11.42	4.72
		Not Predried			14.22	51.72	0.32	42.3	13.55	3.33
		30	248	4.9	14.28	51.32	0.39	40.6	10.61	1.48
		30	248	8.6	14.41	52.03	0.30	42.3	12.35	2.86
		60	248	11.4	14.43	51.83	0.34	40.6	9.64	0.555
		60	248	10.9	14.31	51.55	0.30	41.6	12.05	2.68
		120	248	14.9	14.35	51.52	0.31	41.2	10.82	1.85
		120	248	15.1	14.18	52.12	0.34	41.6	12.95	1.57
		30	302	10.9	14.12	52.83	0.33	42.3	14.15	1.38
		30	302	9.4	13.98	52.97	0.32	42.0	14.03	0.555
		60	302	16.3	14.42	51.43	0.23	42.0	11.75	3.68
		60	302	22.6	14.13	48.55	0.35	42.8	14.74	10.2
		120	302	26.0	14.24	48.23	0.33	40.8	11.11	7.96
		120	302	24.0	14.35	49.17	0.34	40.8	10.53	6.11
		30	356	10.8	13.86	48.49	0.37	40.6	13.21	6.57
		30	356	13.7	14.04	48.42	0.31	41.2	12.61	7.50
		60	356	22.0	14.04	50.09	0.33	40.8	12.31	4.26
		60	356	22.3	14.28	50.45	0.34	40.3	10.24	2.86
		120	356	30.8	14.61	50.27	0.46	41.2	9.35	4.16
		120	356	32.3	14.60	50.52	0.36	40.8	8.74	3.14

(a) Sintered for 60 minutes at 930°F

(b) Analyses before predrying: B = 10.13 per cent C = 32.85 per cent H = 3.57 per cent

(c) Analyses before predrying B = 9.49 per cent C = 30.89 per cent H = 3.94 per cent

TABLE XXII

PREDRYING AND SINTERING DATA FOR BORIC ACID - CARBON GRANULES

Expt. No.	Description of Material	Size Fraction	Sintering Conditions	Weight in g.	Analysis of Product B Per cent	C Per cent	H Per cent	Weight Out g.	B Loss Per cent	C Loss Per cent
RG-138	Powdered BA + C + H_2O Spray	6 mesh - 1/4 in.	Before	--	9.47	30.9	3.74	--	--	--
		6 mesh - 1/4 in.	Predried 1 hr. at 200°F	400						
			Sintered 1 hr. at 1000°F		15.7	49.4	0.57	190	21.4	24.0
		6 mesh - 1/4 in.	Predried 1 hr. at 200°F	400						
			Sintered 1 hr. at 1000°F		16.5	47.2	0.64	190	17.4	27.5
		1/2 in. Plus	Before	--	11.2	35.4	2.79	--	--	--
		1/2 in. Plus	Predried 1 hr. at 200°F	400						
			Sintered 1 hr. at 1000°F		16.0	49.4	0.31	187	33.3	34.7
		1/2 in. Plus	Predried 1 hr. at 200°F	400						
			Sintered 1 hr. at 1000°F		15.9	50.6	0.80	191	32.1	31.8
RG-139	Powdered BA + C + 5 per cent BA Spray	6 mesh - 1/4 in.	Before	--	12.0	37.7	2.41	--	--	--
		6 mesh - 1/4 in.	Predried 1 hr. at 200°F	400						
			Sintered 1 hr. at 1000°F		15.5	40.3	0.83	186	40.0	50.2
		6 mesh - 1/4 in.	Predried 1 hr. at 200°F	400						
			Sintered 1 hr. at 1000°F		15.8	45.0	0.44	183	39.7	45.3
		1/4 in. - 3/8 in.	Before	--	9.0	30.1	3.98	--	--	--
		1/4 in. - 3/8 in.	Predried 1 hr. at 200°F	400						
			Sintered 1 hr. at 1000°F		14.1	51.5	0.43	200	21.7	14.4
		1/4 in. - 3/8 in.	Predried 1 hr. at 200°F	400						
			Sintered 1 hr. at 1000°F		13.9	52.5	0.49	197	24.2	11.5
		3/8 in. - 1/2 in.	Before	--	8.34	32.4	4.10	--	--	--
		3/8 in. - 1/2 in.	Predried 1 hr. at 200°F	400						
			Sintered 1 hr. at 1000°F		15.0	48.9	0.25	219	4.8	17.4
RG-139	Powdered BA + Carbon + 5 per cent BA Spray	3/8 in. - 1/2 in.	Predried 1 hr. at 200°F	400						
			Sintered 1 hr. at 1000°F		14.6	51.4	0.38	197	13.7	21.8
		1/2 in. Plus	Before	--	8.5	29.7	3.95	--	--	--
		1/2 in. Plus	Predried 1 hr. at 200°F	400						
			Sintered 1 hr. at 1000°F		15.0	49.5	0.40	215	4.7	10.4
		1/2 in. Plus	Predried 1 hr. at 200°F	400						
			Sintered 1 hr. at 1000°F		15.2	49.6	0.35	215	3.5	10.2
RG-140	Granular BA + C + H_2O Spray	6 mesh - 1/4 in.	Before	--	8.9	29.1	4.05	--	--	--
		6 mesh - 1/4 in.	Predried 1 hr. at 200°F	400						
			Sintered 1 hr. at 1000°F		15.8	47.6	0.23	195	13.5	20.2
		6 mesh - 1/4 in.	Predried 1 hr. at 200°F	400						
			Sintered 1 hr. at 1000°F		15.8	46.8	0.27	190	15.8	23.6
		1/4 in. - 3/8 in.	Before	--	9.1	29.6	3.77	--	--	--
		1/4 in. - 3/8 in.	Predried 1 hr. at 200°F	400						
			Sintered 1 hr. at 1000°F		17.0	43.7	0.22	189	11.8	30.2
		1/4 in. - 3/8 in.	Predried 1 hr. at 200°F	400						
			Sintered 1 hr. at 1000°F		15.6	48.2	0.20	201	13.8	18.2
		3/8 in. - 1/2 in.	Before	--	10.4	31.4	4.05	--	--	--
			Predried 1 hr. at 200°F	400						
			Sintered 1 hr. at 1000°F		16.3	47.8	0.23	240	7.2	8.8

TABLE XXIII
COMPARISON OF SINTERING LOSSES FOR REGULAR AND PREDRYING SINTERING PROCEDURES

Run No.	Fraction	Per cent Boron Loss (a)		Per cent Carbon Loss (a)	
		Regular	Predried	Regular	Predried
RG-138	-6 Mesh	32.5		32.8	
	6 Mesh 1/4 in.	28.9	19.4	32.0	25.7
	1/4 in. - 3/8 in.	36.7		39.5	
	3/8 in. - 1/2 in.	25.4		27.0	
	+ 1/2 in.		32.7		33.2
RG-139	-6 Mesh				
	6 Mesh - 1/4 in.	37.8	39.9	36.9	47.8
	1/4 in. - 3/8 in.	23.6	23	23.4	13.0
	3/8 in. - 1/2 in.	13.7	9.3	20.0	19.8
	+ 1/2 in.	10.5	4.1	13.8	10.3
RG-140	6 Mesh - 1/4 in.	19.5	14.7	22.3	21.9
	1/4 in. - 3/8 in.	20.7	12.8	19.1	24.2
	3/8 in. - 1/2 in.	27.1	7.2	19.1	8.8

(a) Average values presented for duplicate runs.

TABLE XXIV

VACUUM DRYING OF A-100W BORIC ACID CARBON GRANULES

Drying (a) Conditions		Analysis of Dried Product			
Time hr.	Temperature °F	B Per cent	C Per cent	H Per cent	B:H (b) Ratio
2	138	11.3	38.9	3.38	3.34
2	259	12.1	36.6	2.66	4.55
2	318	14.2	42.4	1.03	13.8
2	405	14.2	50.4	0.89	16.0
1	140	9.3	36.7	3.98	2.34
2	140	10.8	32.8	3.71	2.91
4	140	10.0	31.0	4.59	2.18
1	194	10.6	34.3	3.43	3.09
2	194	10.3	36.1	3.67	2.81
4	194	11.2	36.2	3.37	3.33

(a) All tests made under vacuum of 23-25 inches Hg.

(b) B:H ratio in H_3BO_3 = 3.58

TABLE XXV
EFFECT OF SINTERING METHODS ON DEGRADATION OF GRANULAR FEED

Identification of Material	Description of Material (Processing Conditions)	Sintering Conditions (a)	Amount of Degradation Per cent
RG-138	Powdered BA. + carbon + water spray	1 hour at 200°F + 1 hour heat-up to 1000°F + 1 hour at 1000°F	14.3
RG-138	Powdered BA. + carbon + water spray	1 hour at 1000°F	13.2
RG-138	Powdered BA. + carbon + water spray	1 hour at 200°F in drying oven 1 hour at 1000°F in sinter oven	33.4
RG-139	Powdered BA. + carbon + BA. spray	1 hour at 200°F + 1 hour heat-up to 1000°F + 1 hour at 1000°F	4.1
RG-139	Powdered BA. + carbon + BA. spray	1 hour at 1000°F	6.8
RG-139	Powdered BA. + carbon + BA. spray	1 hour at 200°F in drying oven 1 hour at 1000°F in sinter oven	18.8
RG-140	Granular BA. + carbon + water spray	1 hour at 200°F + 1 hour at 1000°F + 1 hour heatup to 1000°F	8.7
RG-140	Granular BA. + carbon + water spray	1 hour at 1000°F	2.1
RG-140	Granular BA. + carbon + water spray	1 hour at 200°F in drying oven 1 hour at 1000°F in sinter oven	7.8

(a) 1/2 in. loading on 8 x 13 x 1/2 in. stainless steel trays

TABLE XXVI
CARBON AND BORON LOSSES
FROM BORIC ACID-CARBON GRANULES EXPOSED TO HOT AIR OR FLUE GAS

Description of Gas			Gas. Temp. °F	Average Bed Temp. °F	Run Time hr.	Losses, per cent		
Gas	Composition	Per cent				Over-all	B	C
Air (Dry)	N_2	77.9	1120	862	1	46.7	3.7	11.3
	O_2	22.1	1160	806	2	45.4	0.0	9.6
Air (Wet)	N_2	68.4	1200	982	1	67.2	26.2	59.4
	O_2	21.8	1200	1022	2	72.3	30.1	72.1
	H_2O							
Flue Gas (Wet)	N_2	74.8	1220	951	1	53.0	27.3	10.7
	CO_2	15.0	1160	872	2	54.2	36.5	6.5
	H_2O	10.2	795	710	2	60.7	59.5	6.9
			1000	850	2	58.2	48.9	8.6
Flue Gas (Wet)	N_2	70.2	1170	943	1	54.9	28.5	13.1
	CO_2	14.9	1240	1011	2	74.2	48.3	63.1
	H_2O	10.2	805	697	2	57.8	51.9	5.2
	O_2	4.7	988	842	2	58.0	49.7	14.8

TABLE XXVII
ROTARY CALCINER OPERATING DATA

Expt. No.		C-1	C-2	C-3	C-4	C-5	C-6	C-7
Residence Time in Hot Zone	hr.	0.23	0.4	0.20	0.20	0.27	0.20	0.23
Calciner Speed	r.p.m.	1.20	1.20	1.20	1.25	1.25	1.25	1.25
Calciner Slope	in./ft.	0.14	0.28	0.28	0.28	0.28	0.28	0.28
Feed Formulation		59.2W	40.8 SuWa(63Wa) →			A150W 25.3 SuWa(70Wa)		
Total Charge	lb.	50	50	50	50	50	50	50
Total Back-up	lb.	4.0	0.3	(a)	1.0	(a)	--	9.9
Total Product	lb.	29.0	29.9	---	29.8	---	20.4	23.4
Sintering Losses	per cent	37.0	39.8	---	39.2	---	59.2	44.1
Equilibrium Temp.	°F	750 (b)	1206	1290	1380	1100	1100	1188
Feed Rate	lb./hr.	11.3	6.3	---	14.3	---	11.8	13.0
Back-up Rate	lb./hr.	0.9	neglig.	---	0.3	---	---	2.6
Product Rate	lb./hr.	6.6	4.1	---	6.6	---	4.8	6.5
Product Analysis	per cent							
B		---	0.05-0.7	---	0.02-0.05	12.0-13.0	11.3-13.1	8.7-11.6
C		---	95.5-99.2	---	83.5-99.6	54.7-58.9	52.4-56.6	42.5-56.7
H		---	0.39-0.62	---	0.39-1.83	0.35-0.36	0.3-1.04	0.71-3.0

(a) Data incomplete due to short run caused by plug in feeder.
(b) Improperly located thermocouple.

TABLE XXVIII
OPERATING CONDITIONS AND RESULTS FROM TWO STAGE DRYING TESTS AT GENERAL AMERICAN TRANSPORTATION CORPORATION

Test Number	First Stage Tests							Second Stage	
	1	2	3	4	5	6	7	$P_1 + P_2$ 1	P_7 2
Feed, lb/hr.	60	120	120	60	60	60	45	60	45
Feed, H_2O Per cent, Wet Basis	20.9	20.9	19.0	19.0	18.6	18.6	19.1	0	2.8
Product H_2O Per cent, Wet Basis	0	0	10.0	8.1	7.9	0	0	0	0
Feed Temp., °F	75	75	75	75	75	75	75	75	75
Product Temp., °F	400	240	210	240	174	180	220	1060	1080
Inlet Air Temp., °F Dry Bulb	945	950	700	720	850	910	500		
Inlet Air Temp., °F Wet Bulb									
Outlet Air Temp., °F Dry Bulb	450	410	250	250	226	236	244	420	510
Air Velocity, ft./min.	175	175	175	175	175	175	175		
Density of Feed, lb./ft.^{3}	45.0	45.0	45.0	45.0	45.0	45.0	45.0	40.0	
Density of Product, lb./ft.^{3}	40.0							35.0	
Slope of Dryer	0	0	0	0	0	0	0	0	0
R. P. M. of dryer	6	6	6	6	6	6	6	6	6
Length of Test, min.	60	40	60	63	75	30	130	75	75
Retention Time, min.	50							35	50
Knocker	yes	yes	yes	yes	yes	yes	yes	no	no
Fillage								30.0	31.0
Discharge Ring, in.	2	2	2	2	2	2	2	2	2
Stack Temp., °F								1000	1000

TABLE XXIX
SUMMARY OF DRYING AND SINTERING DATA
PROCTOR AND SCHWARTZ DRYING TESTS

Test No.	Test Conditions (a) Dry Bulb Temp. °F	Wet Bulb Temp. °F	Weight Loss During Drying per cent	Analysis of Dried Granules B per cent	C per cent	H per cent	Loss During Drying B per cent	C per cent	Final H:B Ratio (b)	Free Moisture H_2O per cent	Time of Drying Test min.
Feed Material	---	---	---	8.90	27.8	4.20	---	---	0.472	15.4	
1	200	100	25.5	11.4	35.7	2.83	4.6	4.3	0.248	- 3.2	60
2	300	120	27.4	11.3	36.5	2.89	7.6	4.7	0.256	- 2.4	10
3	180	95	30.9	12.1	38.1	2.68	6.1	5.4	0.221	- 6.3	30 + 15 = 45
4	250	150	26.8	10.9	38.2	2.80	10.3	0.0	0.261	- 2.2	30
5	300	180	25.7	11.0	34.7	3.06	8.2	7.2	0.278	- 0.2	20

(a) Air Velocity = 250 ft./min. upflow through perforated tray loaded to 1-1/2 inch depth
(b) Theoretical H-B ratio for H_3BO_3 = 0.280

Qualitative Results On Hardness and Stability Tests On Dried (by P + S) and Sintered Granules

Product from P + S Test Number	Relative (c) Hardness 1 (Best)	Amount of (d) Degradation 1 (Best)
1	5	3
2	2	2
3	3	4
4	4	5
5		

(c) Determined by thumb-finger pressure
(d) Fracturing in reverse order of amounts observed

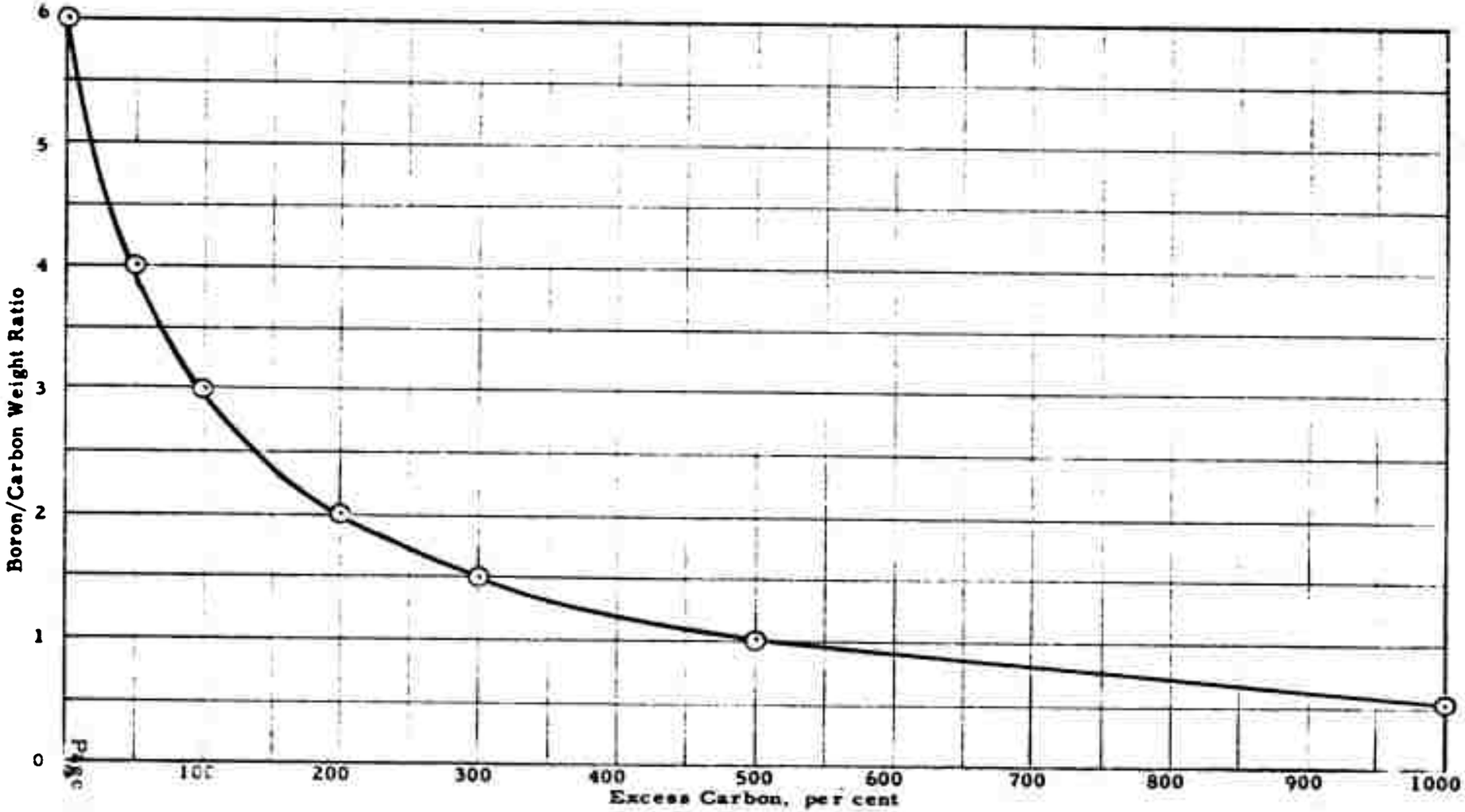


Figure 1 - Boron-Carbon Ratio in Feed Vs. Excess Carbon Used in Feed Formulation Code

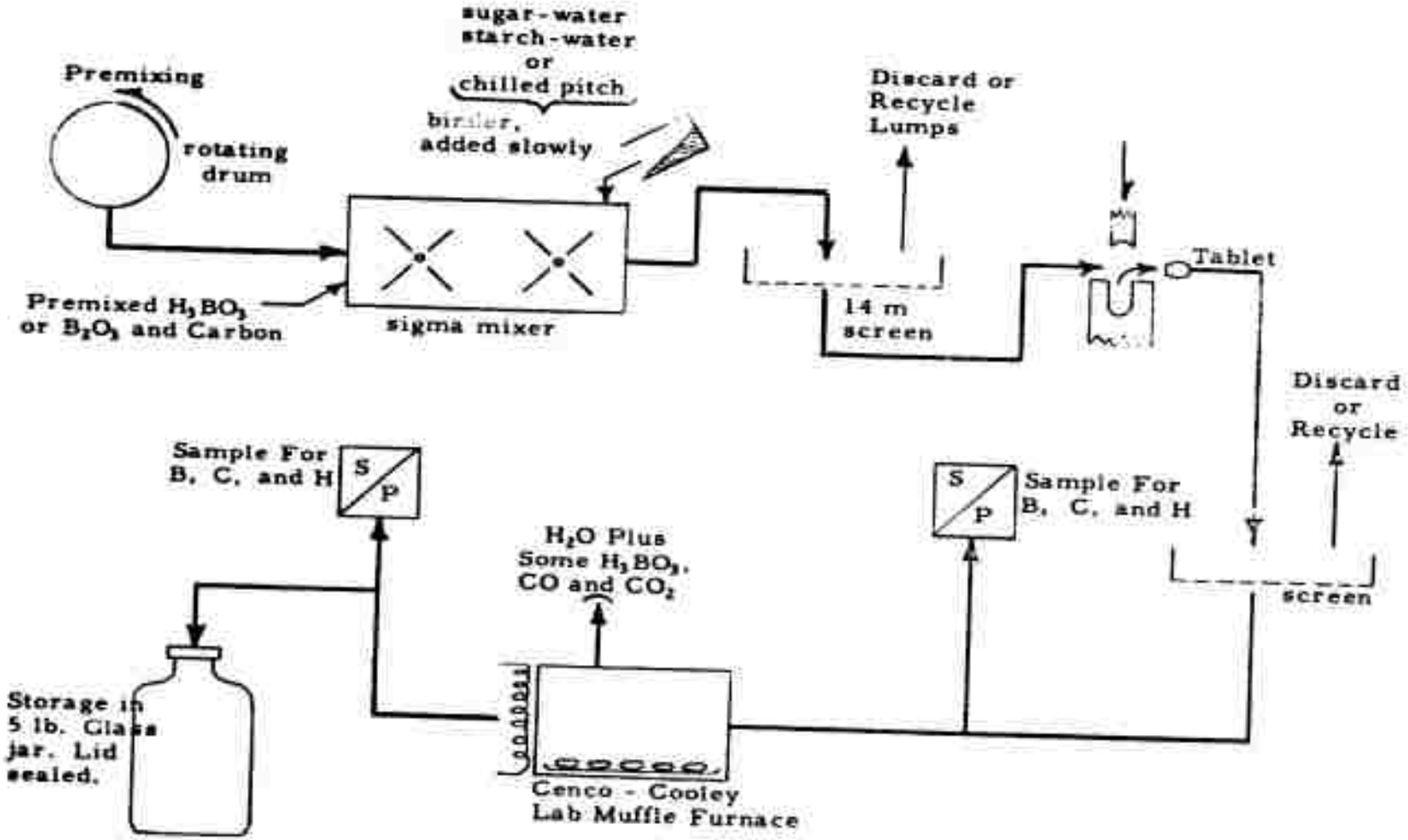


Figure 2A - Process Flow Sheet: Bench Scale and Pilot Plant Feed Preparation Tabletted Feed

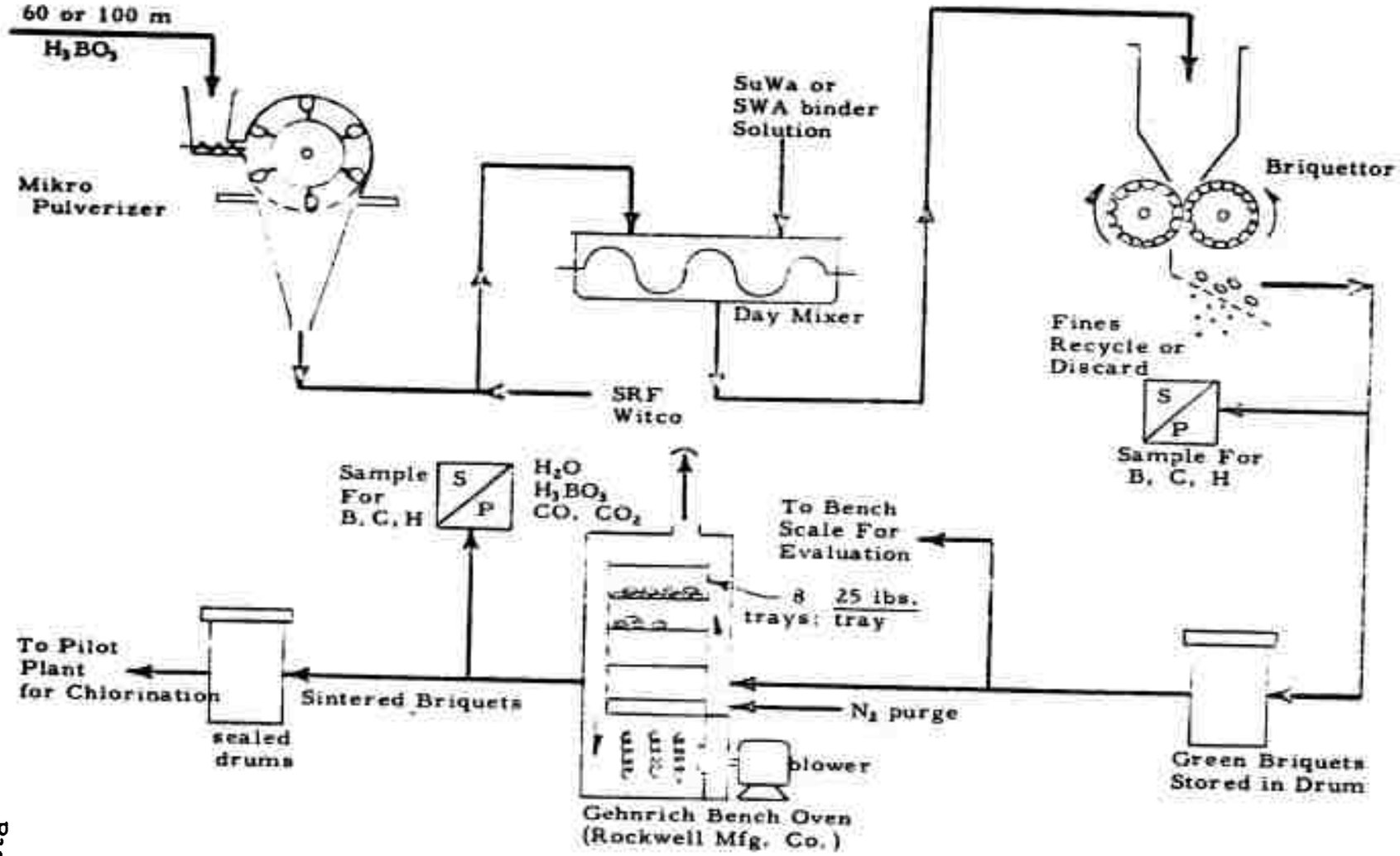


Figure 2B - Process Flow Sheet: Bench Scale and Pilot Plant Feed Preparation
Briquetted Feed

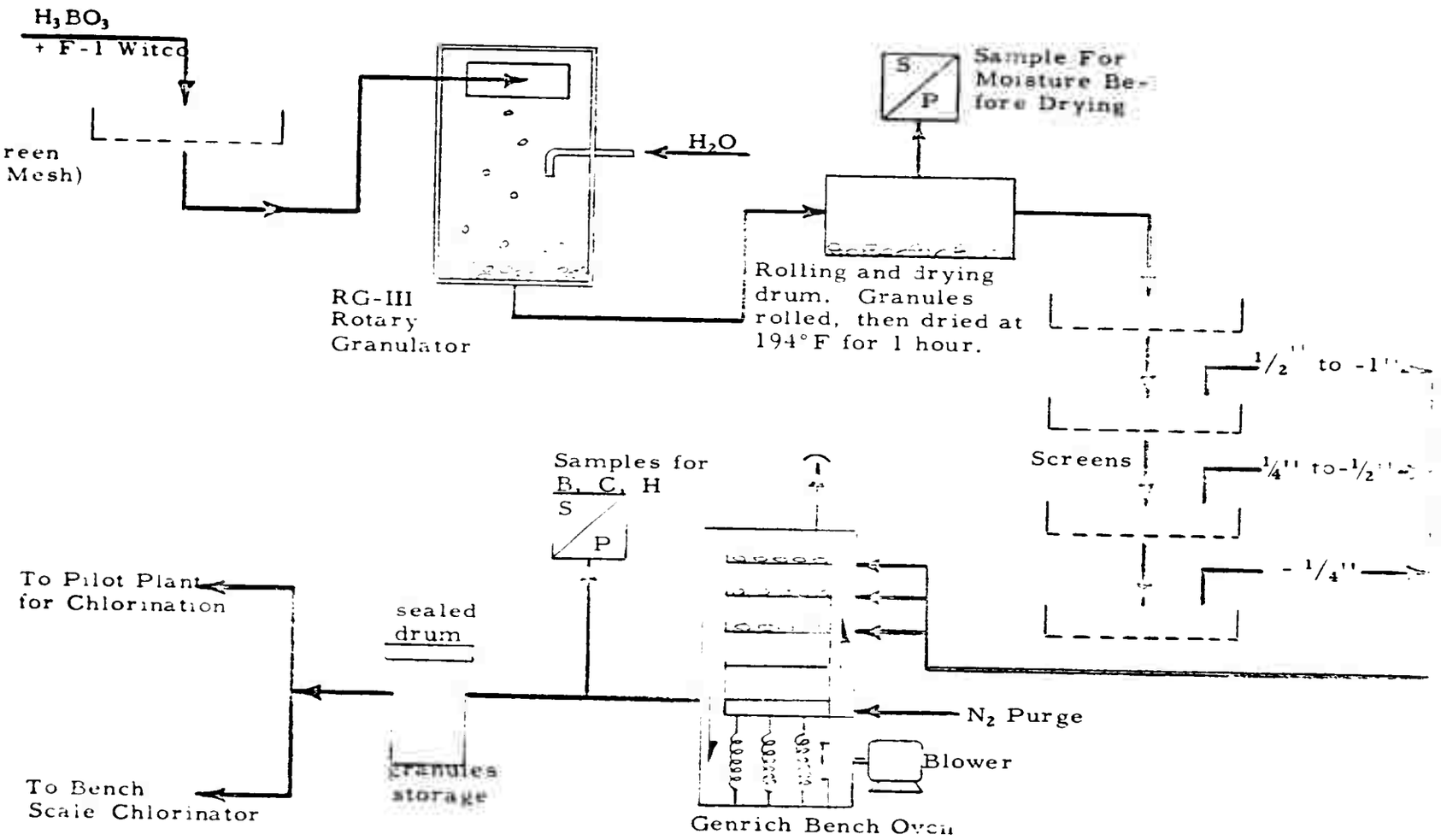
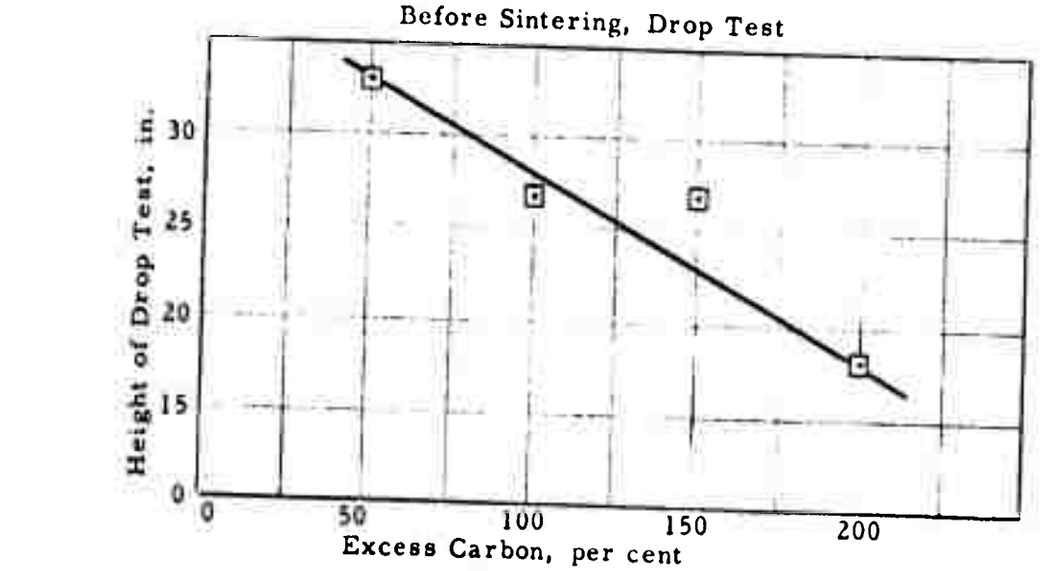


Figure 2C - Process Flow Sheet: Bench Scale and Pilot Plant Feed Preparation
Granulated Feed (Granules) Equipment and Procedure

Excess Carbon per cent	Height of Drop Test in.	Condition of Pellets	
		Before Sintering	After Sintering
50	33	Firm	Slightly Soft
100	27	Firm	Slightyl Soft
150	27	Firm	Hard
200	18	Firm	Soft (Friable)



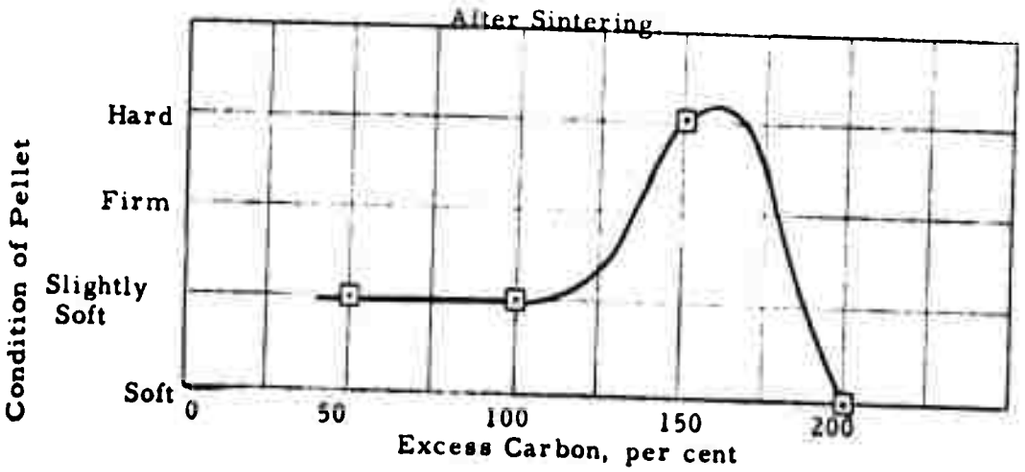


Figure 3 - Effect of Excess Carbon on Pellet Strength
Boric Oxide Pellets with 25 per cent Koppers
150°F Pitch as Binder

Excess Carbon per cent	Drop Test Height in.	Condition of Pellets	
		Before Sintering	After Sintering
20	20	Weak	Slightly Soft
100	21	Fiar	Slightly Soft
150	22	Fair	Hard
200	Very Low	Very Weak	

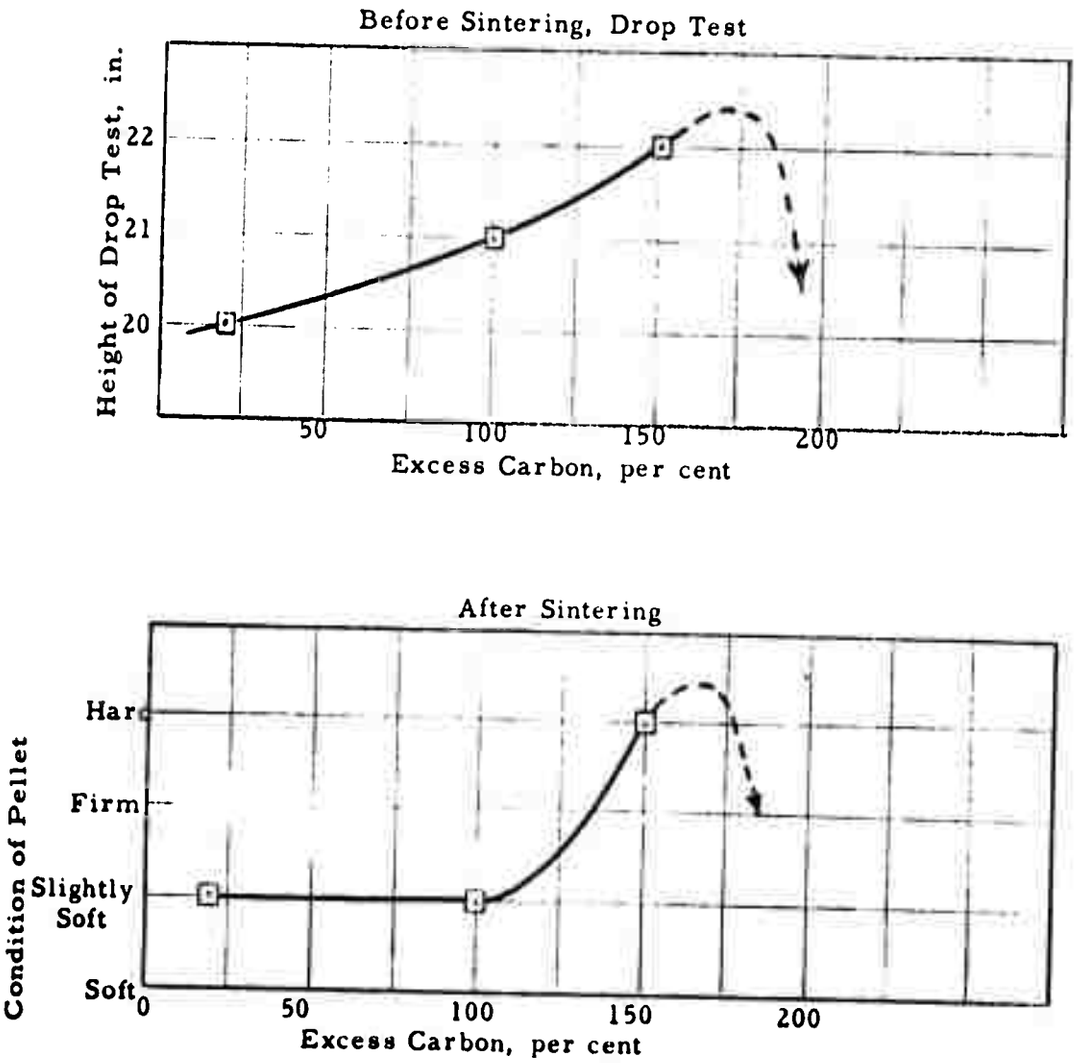
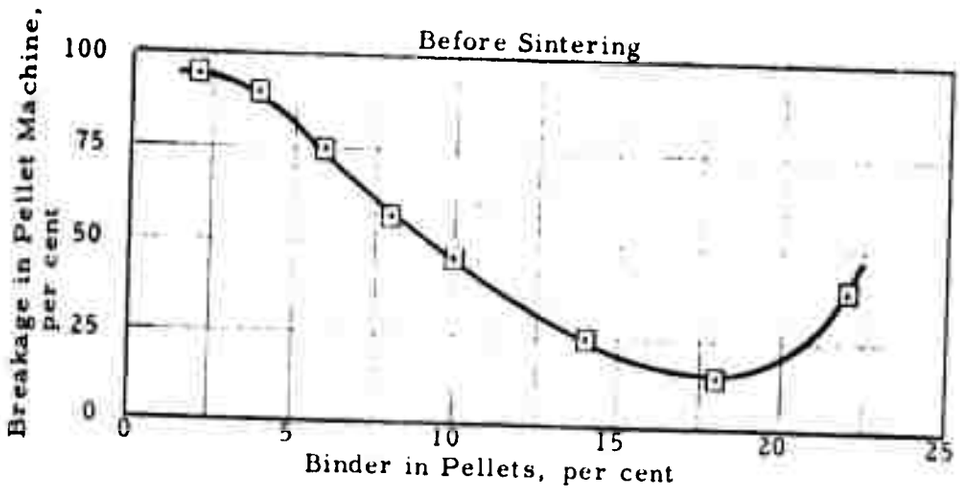


Figure 4 - Effect of Excess Carbon on Pellet Strength
Boric Oxide Pellets with 25 per cent Koppers
200°F Pitch as Binder

Binder per cent	Strength of Pellets	
	Before Sintering	After Sintering
2	95 per cent Broke in Pellet Machine	Soft, Fused, and Deformed
4	90 per Cent Broke in Pellet Machine	Soft, Fused, and Deformed
6	75 per cent Broke in Pellet Machine	Soft, Fused, and Deformed
8	57 per cent Broke in Pellet Machine	Soft, Fused, and Deformed
10	47 per cent Broke in Pellet Machine	Soft, Fused, and Deformed
14	25 per cent Broke in Pellet Machine	Soft
18	15 per cent Broke in Pellet Machine	Firm
22	40 per cent Broke in Pellet Machine	Very Soft



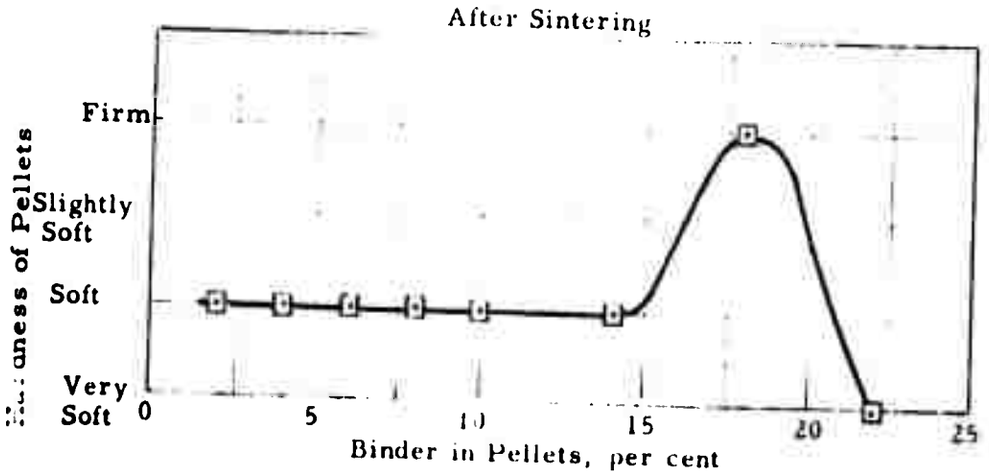


Figure 5 - Effect of Binder on Pellet Strength
Boric Acid Pellets with 10 per cent
Excess Carbon-K-150 Pitch Used as Binder

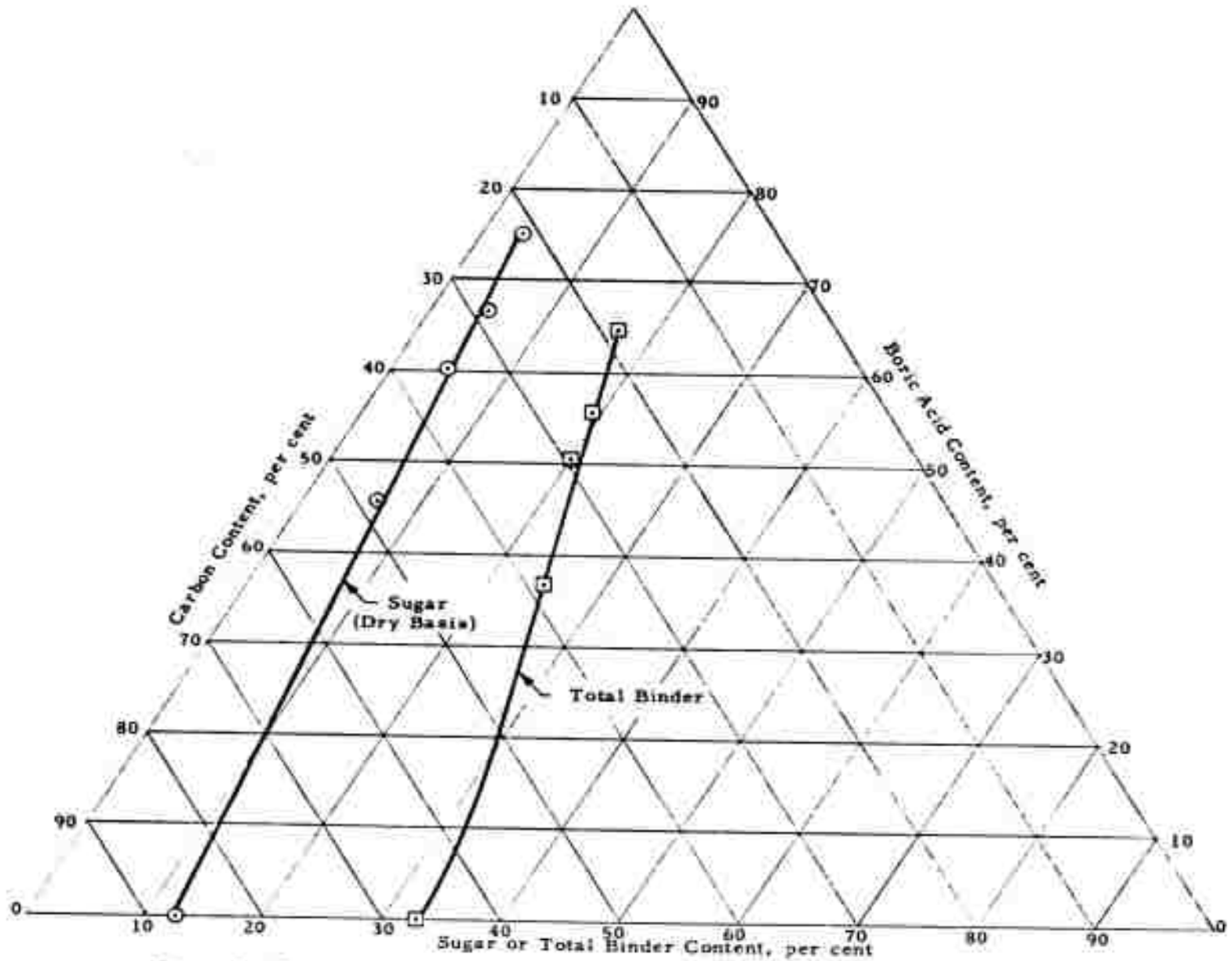


Figure 6 - Optimum Sugar-Water Binder Contents for Boric Acid-Carbon Tablets

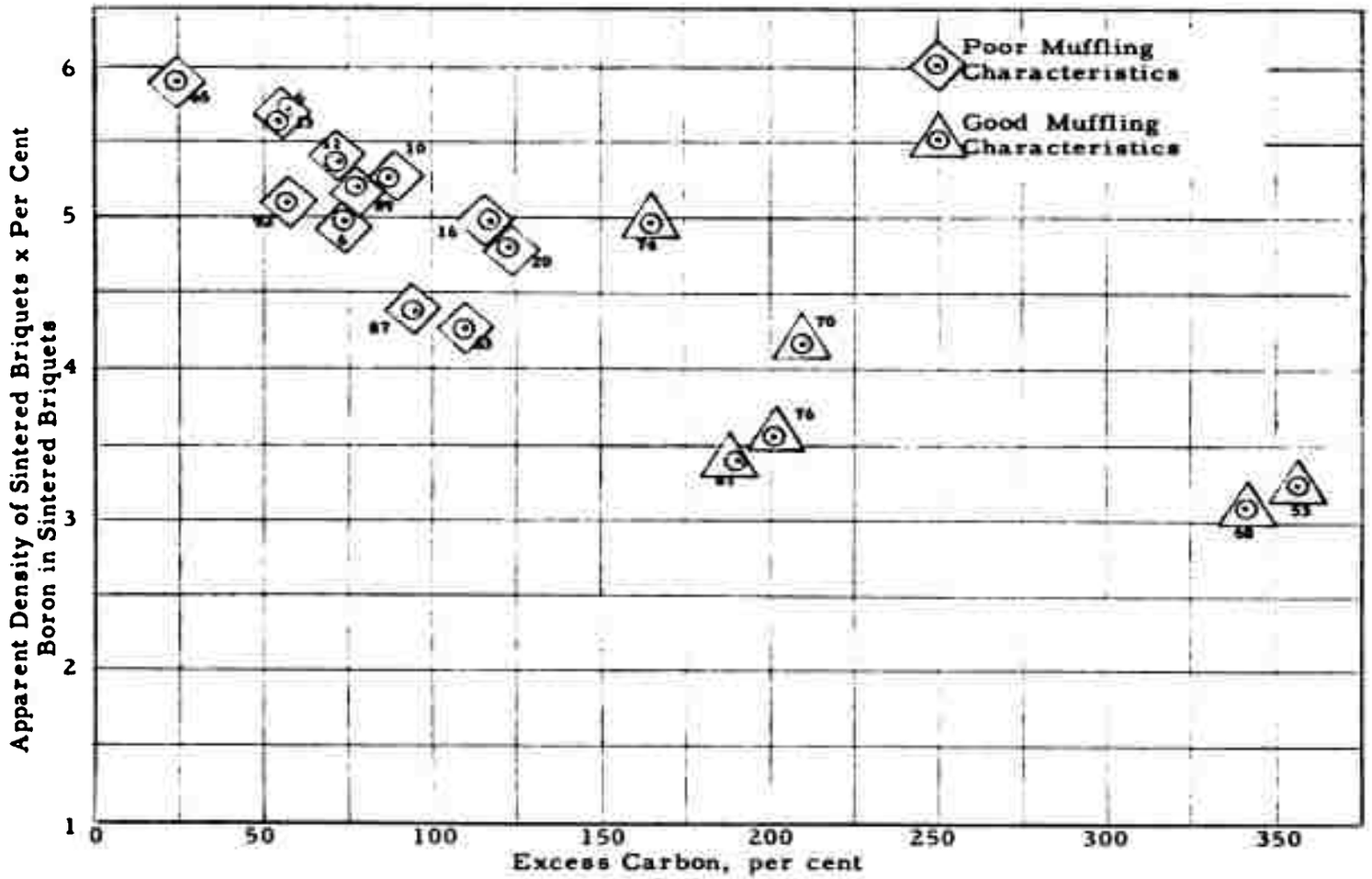


Figure 7 - Briquet Muffle Test Results as Function of Boron Density and Excess Carbon Content in Sintered Briquets

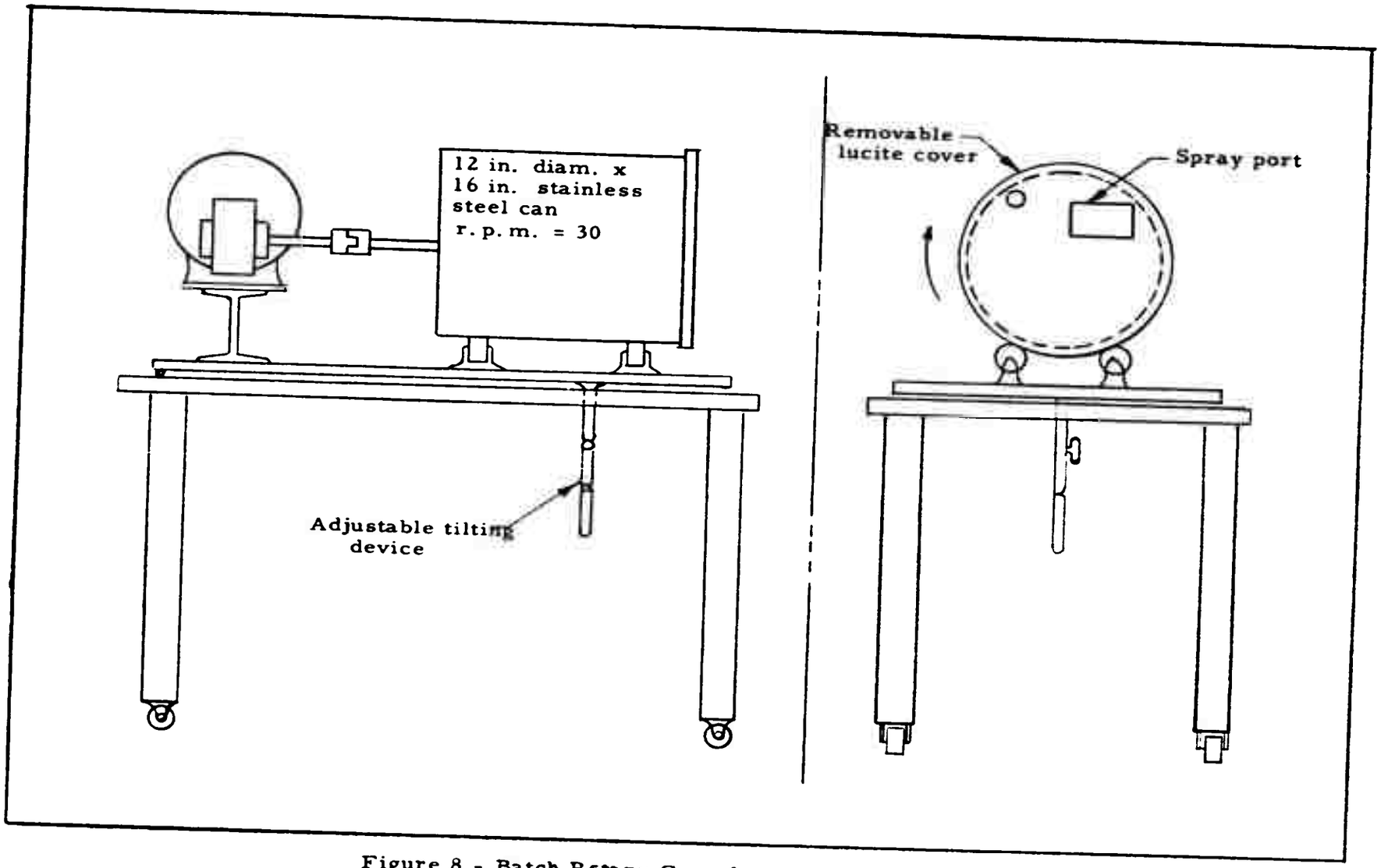
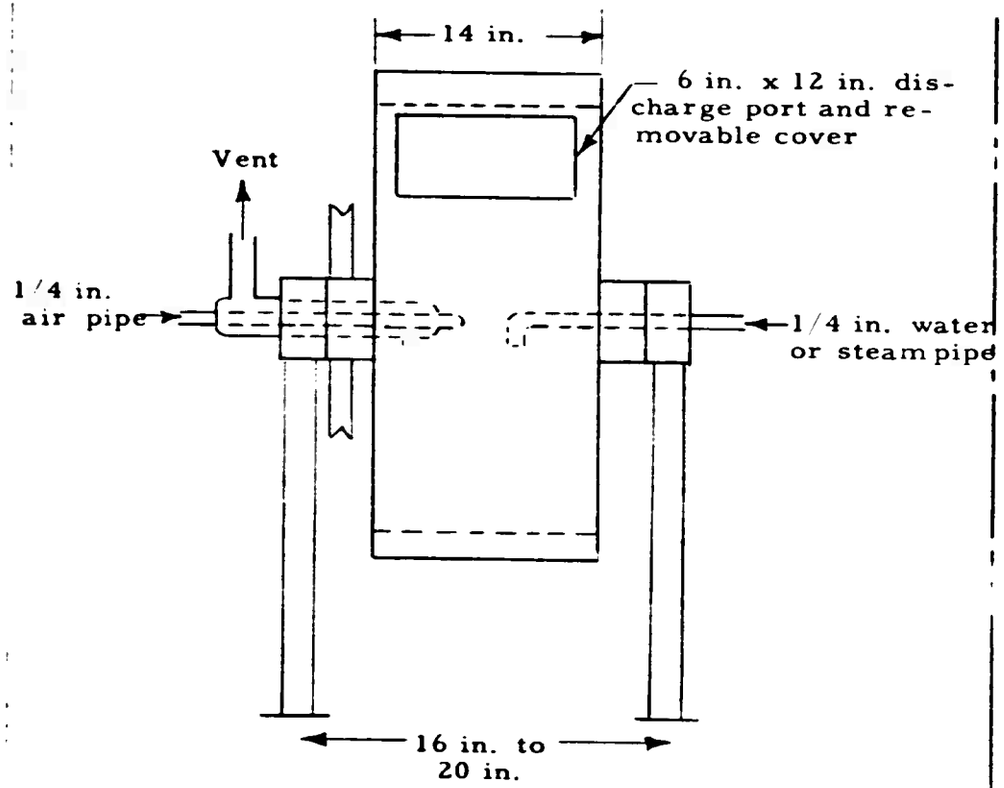
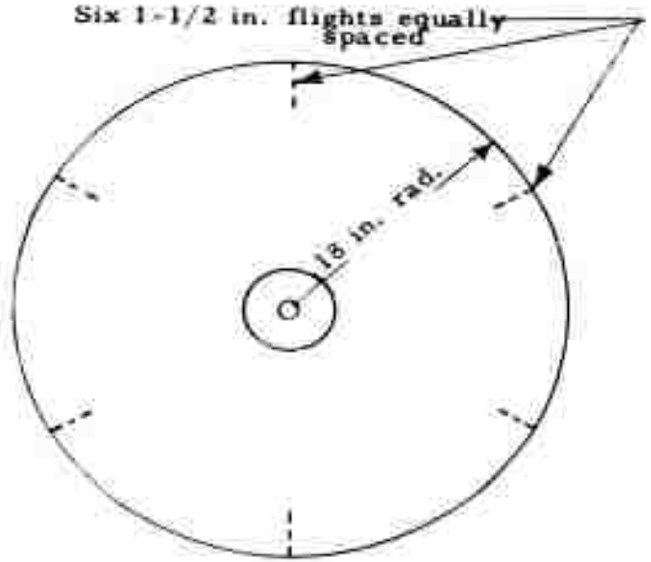


Figure 8 - Batch Rotary Granulator RG-II



Motor to be mounted on support to allow clearance for discharge pan.



Mildsteel drum, 36 in. diam. x 14 in.
r.p.m. = 4 to 10
Batch weight (material) = 40 - 50 lb.

Figure 9 - Batch Rotary Granulator, RG-III

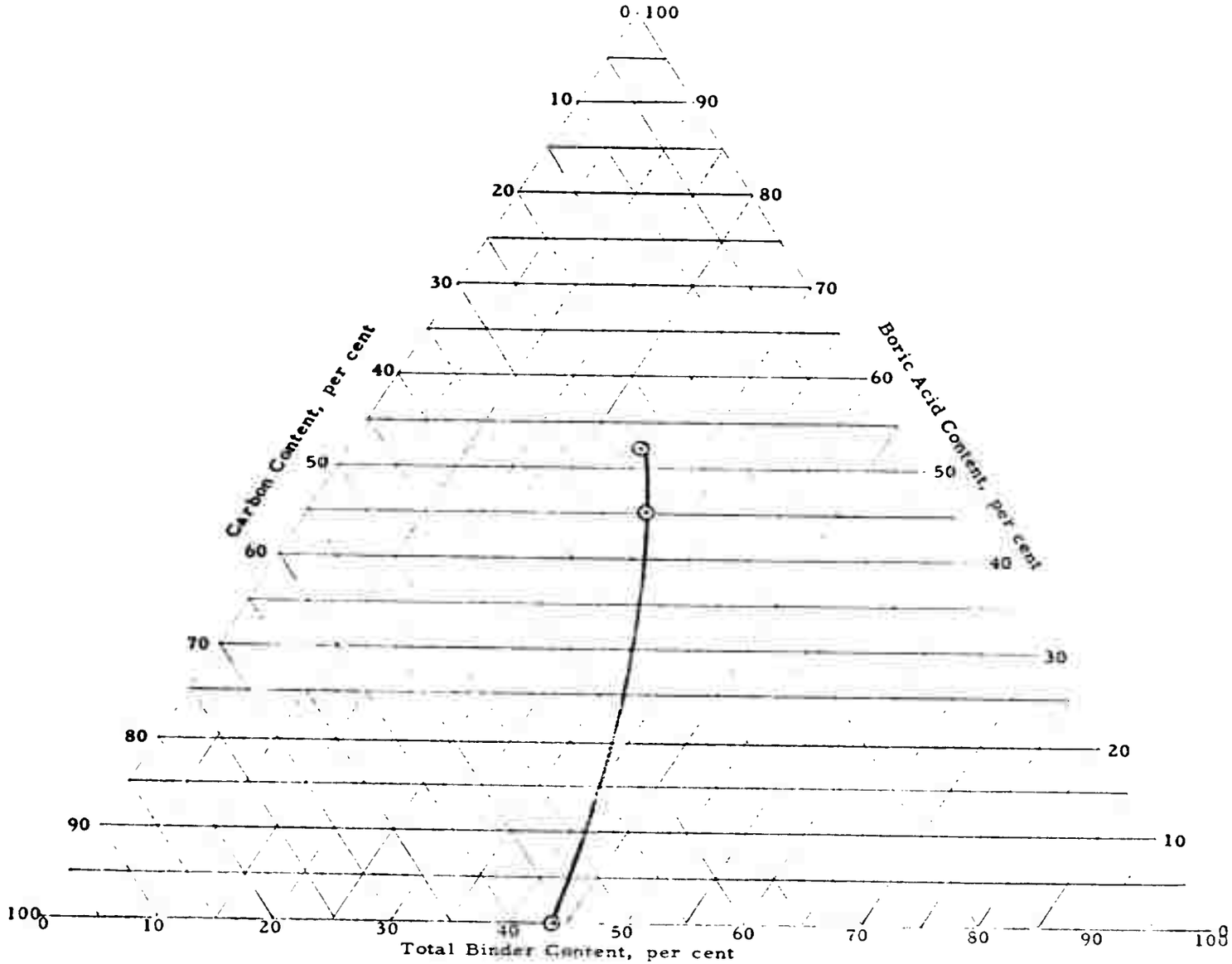


Figure 10 - Optimum Binder Content for Carbon and Boric Acid-Carbon Granules

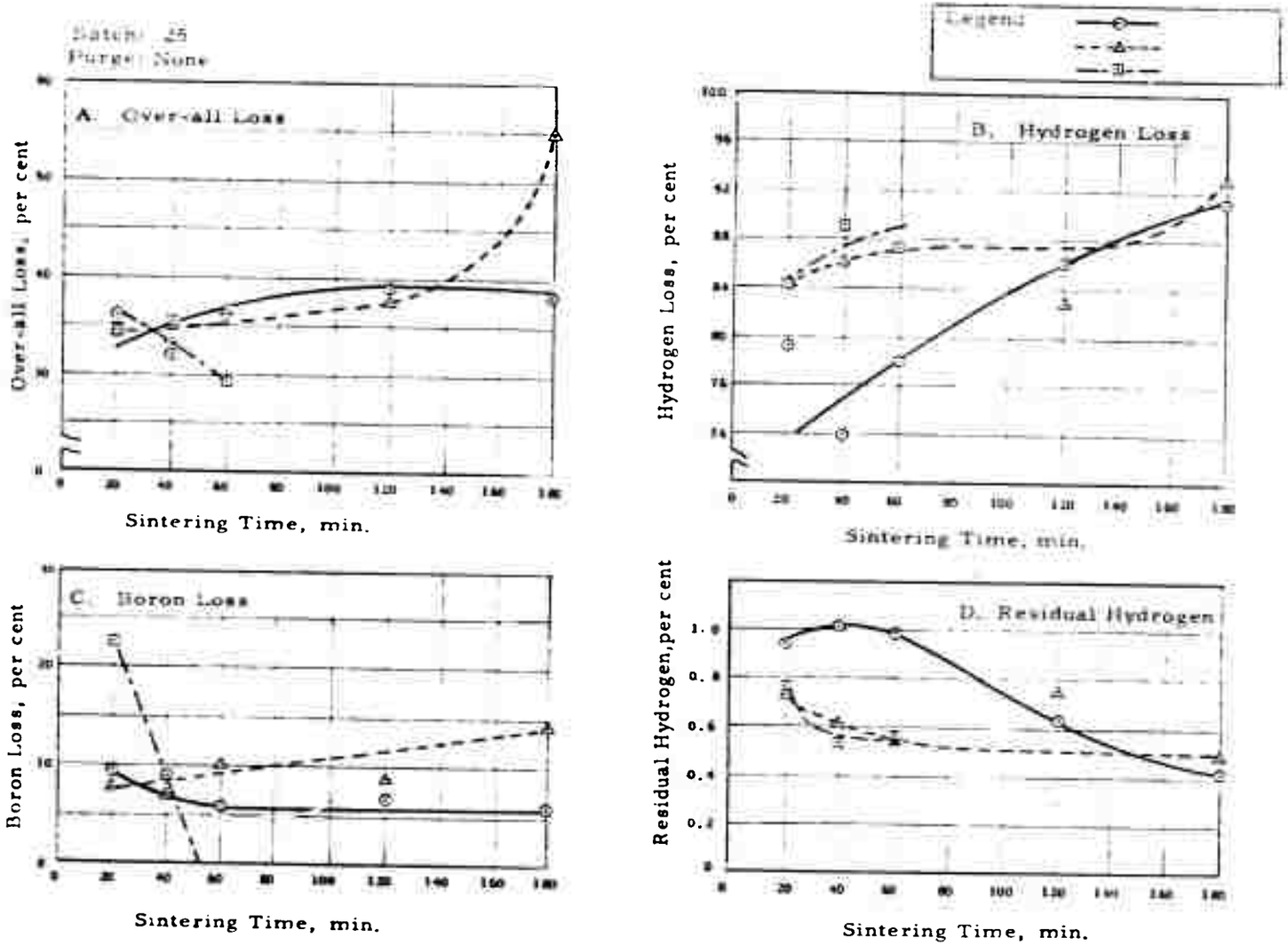


Figure 11. Effect of Sintering Time and Temperature on Over-all, Boron, Carbon, and Hydrogen Losses of A. 100W 20 K150 Tablets.

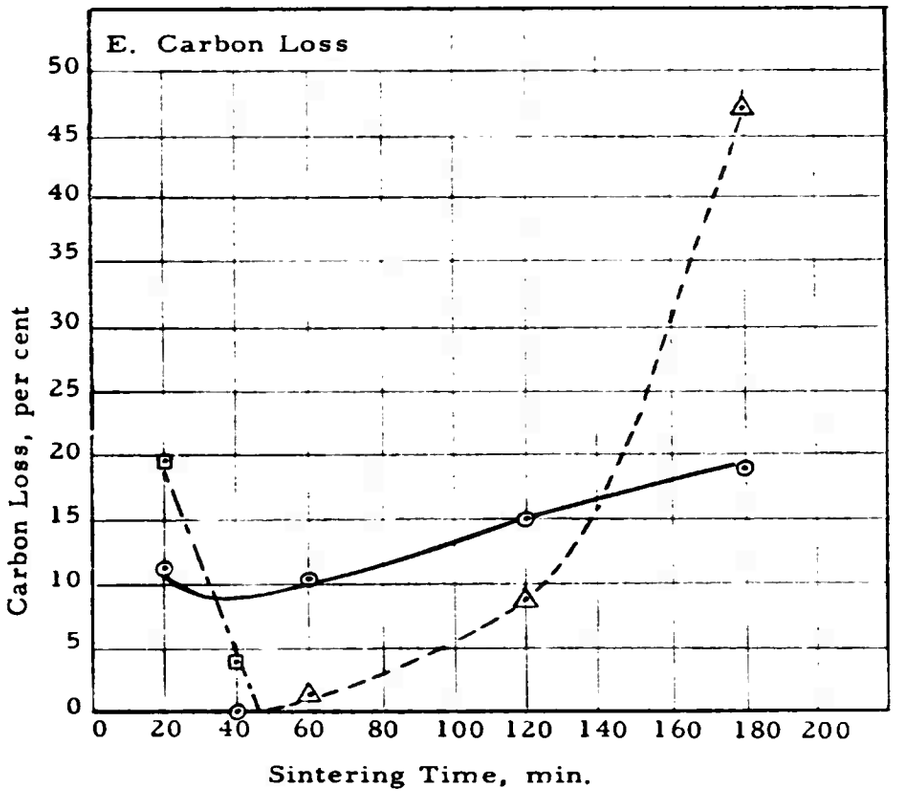


Figure 11 Effect of Sintering Time and Temperature on Over-all, Boron, Carbon, and Hydrogen Losses of A. 100W 20 K150 Tablets (Continued)

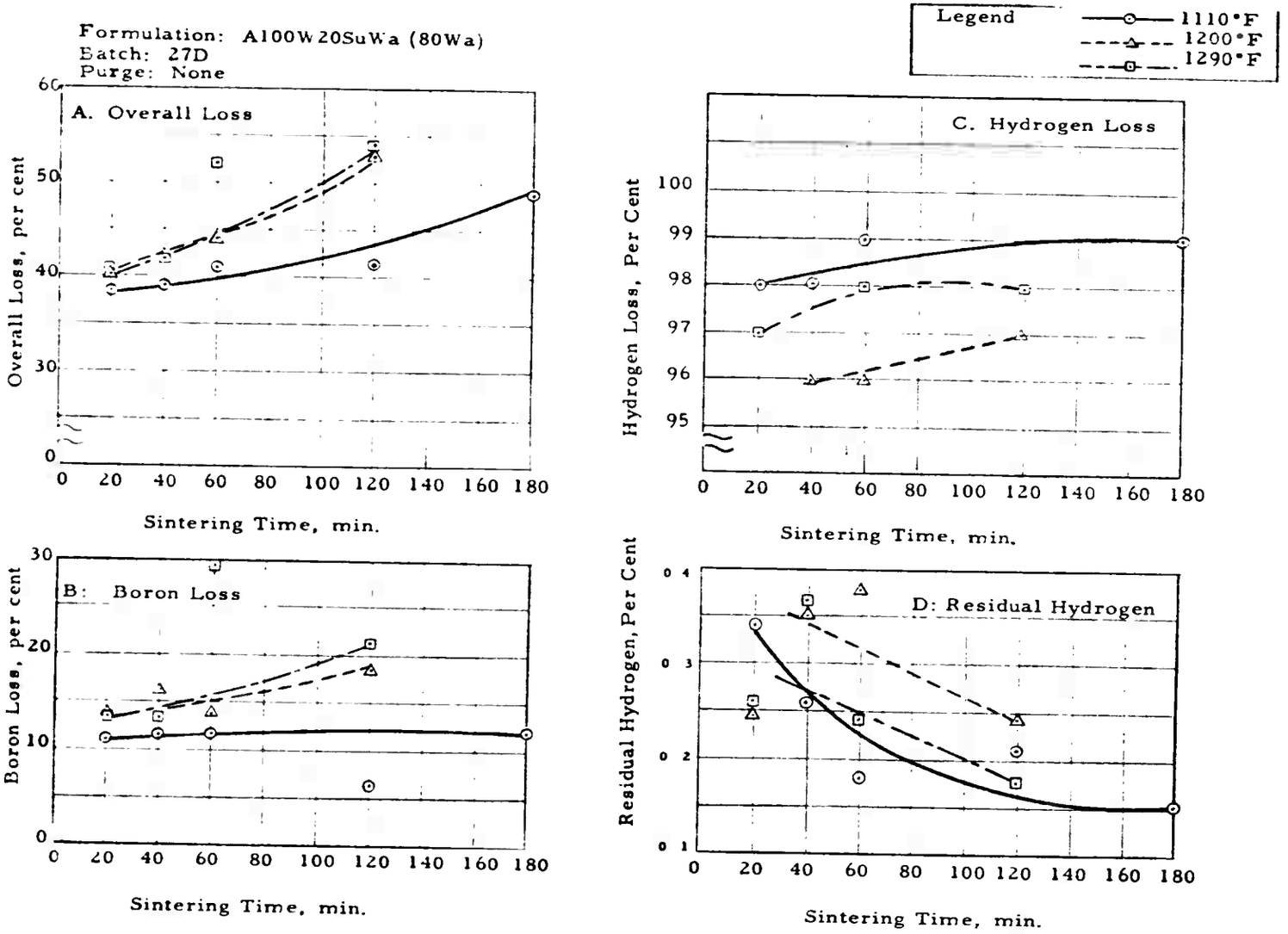


Figure 12A Effect of Sintering Time and Temperature on Boron, Carbon, and Hydrogens Losses of Sugar-Water Bound Tablets

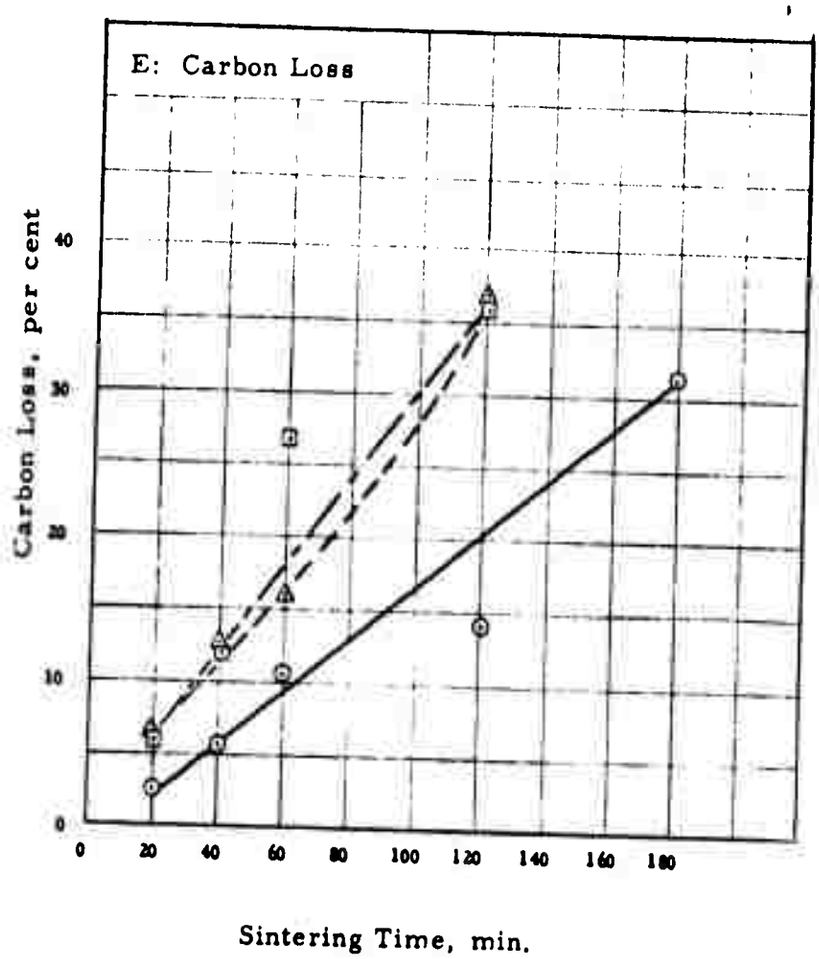


Figure 12A Effect of Sintering Time and Temperature on Boron, Carbon, and Hydrogens Losses of Sugar-Water Bound Tablets (Continued)

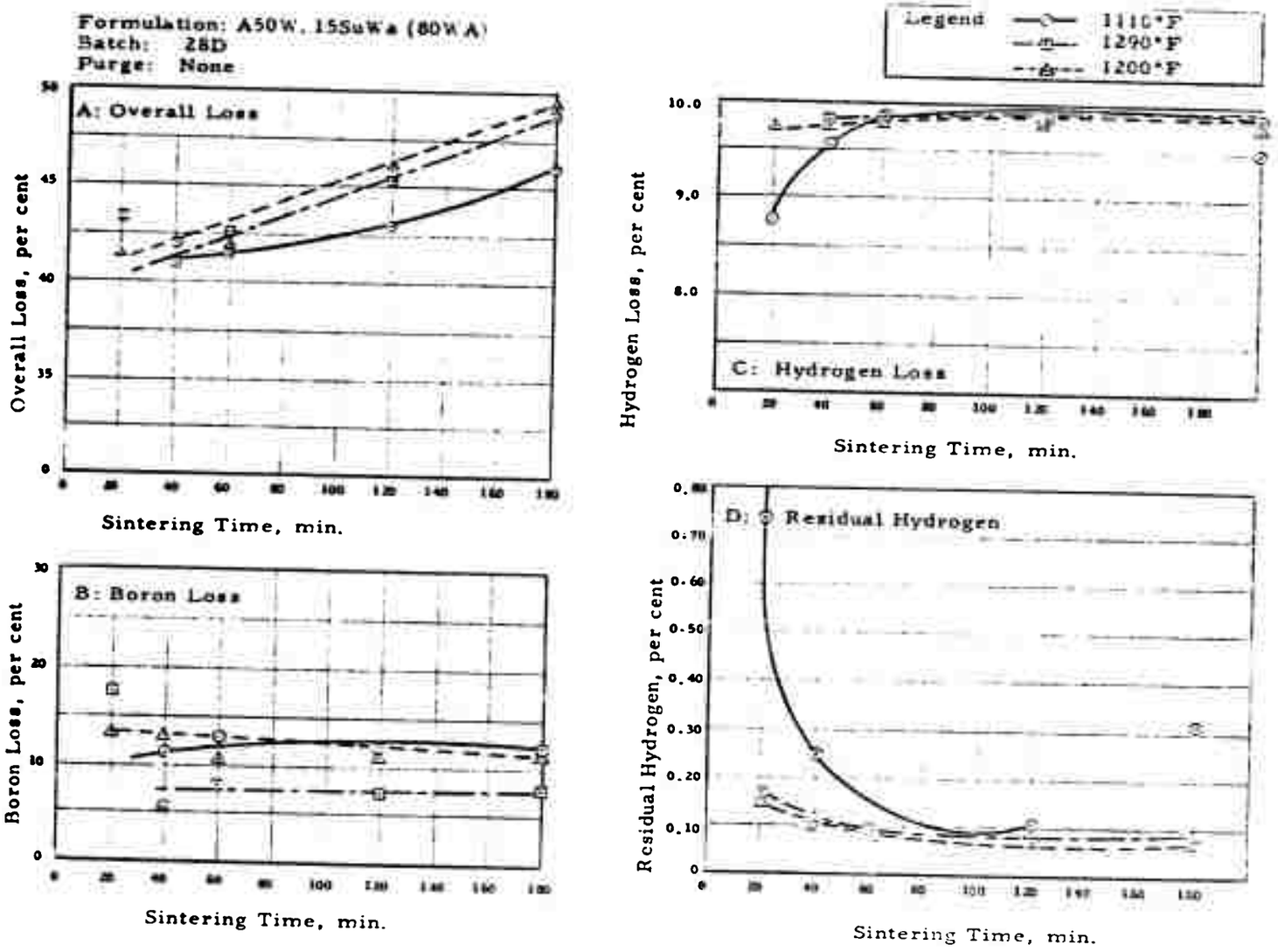


Figure 12B. Effect of Sintering Time and Temperature on B, C, and H. Losses of Sugar-Water Found Tablets.

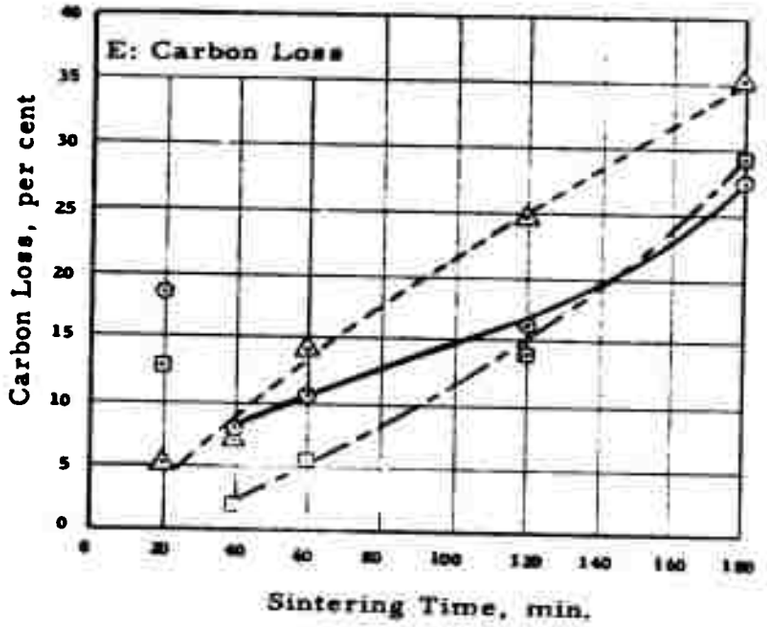


Figure 12B Effect of Sintering Time and Temperature on B, C, and H. Losses of Sugar-Water Bound Tablets. (Continued)

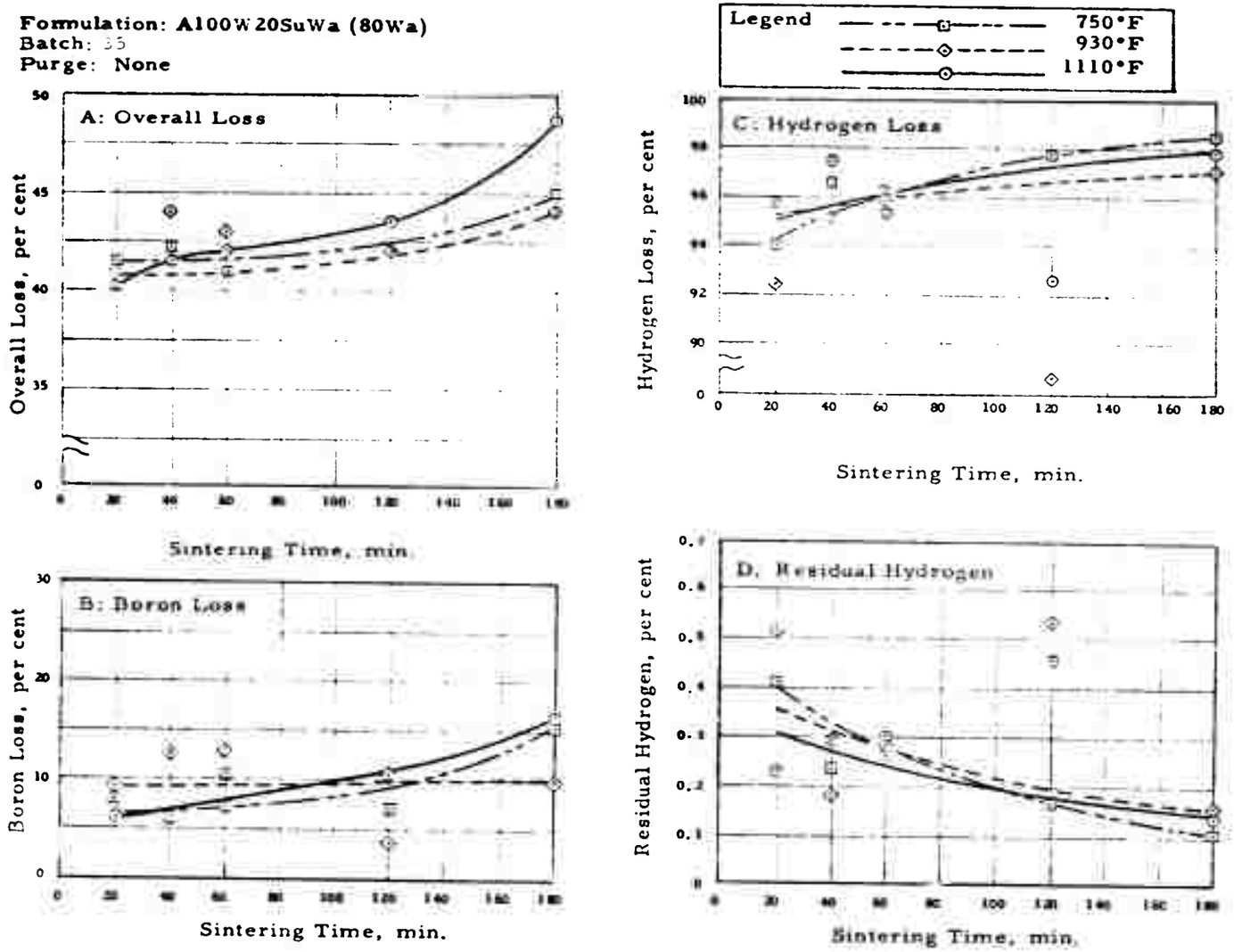


Figure 12 C Effect of Sintering Time and Temperature On B, C and H Losses of Sugar-Water Bound Tablets.

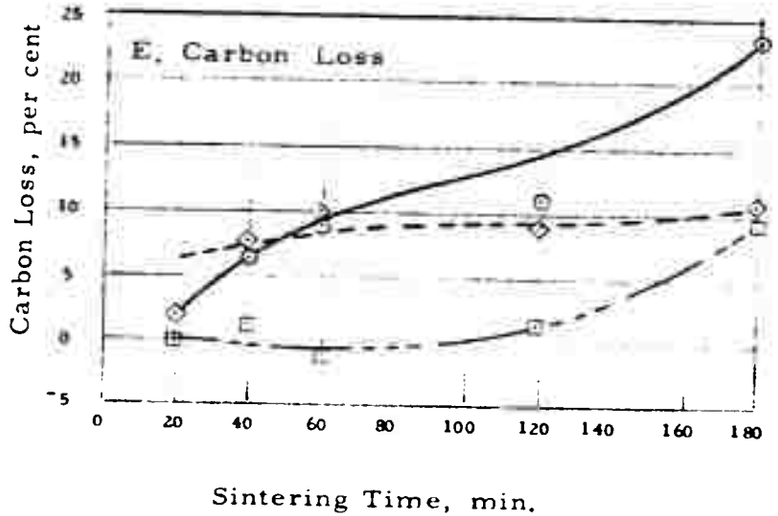
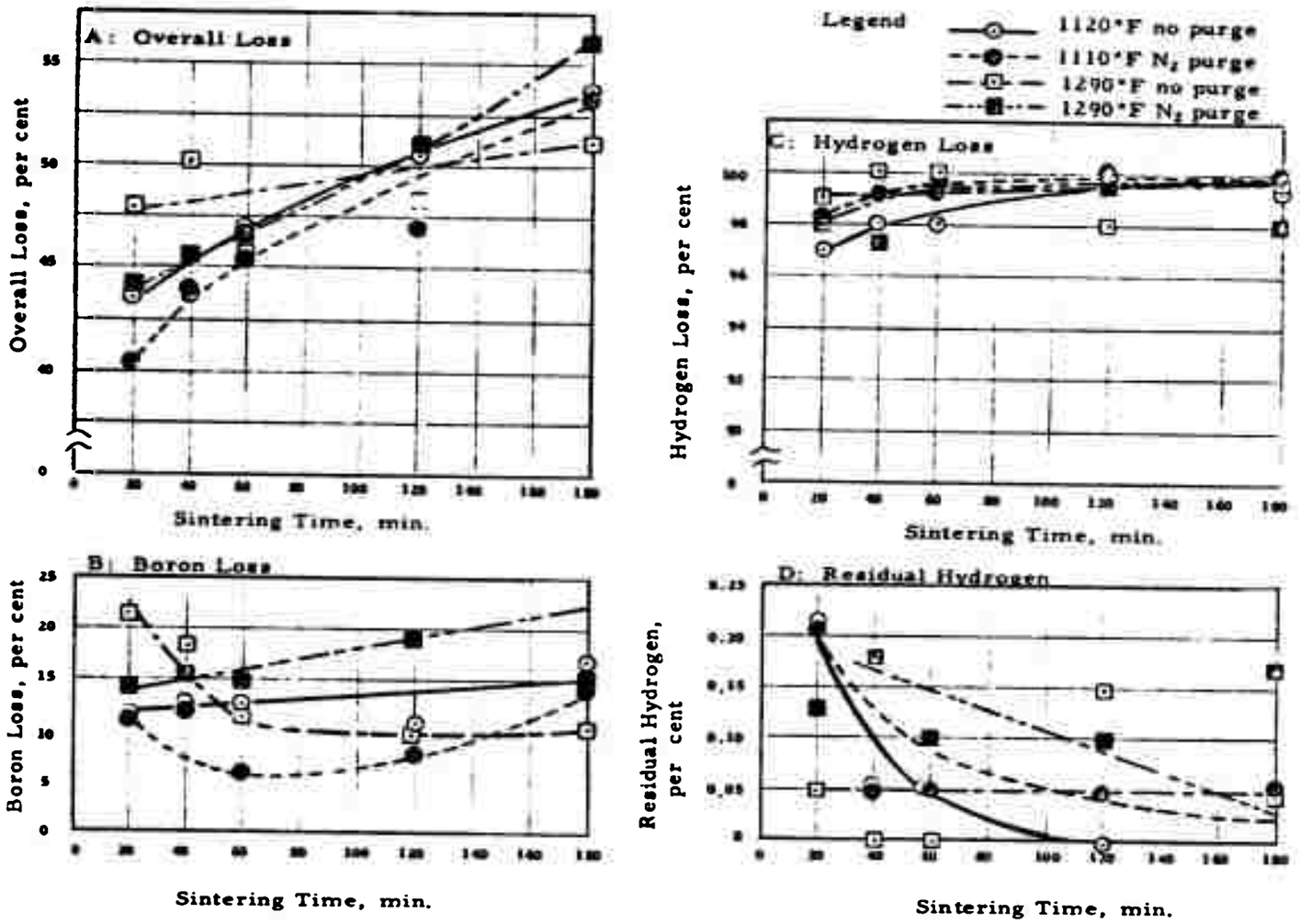


Figure 12C Effect of Sintering Time and Temperature On B, C and H Losses of Sugar-Water Bound Tablets. (Continued)



Formulation: A100W20SuWa (80Wa)
Batch: 39
Purge: None vs. N_2 purge

Figure 12D Effect of Sintering Time and Temperature on B, C and H Losses of Sugar-Water Bound Tablets

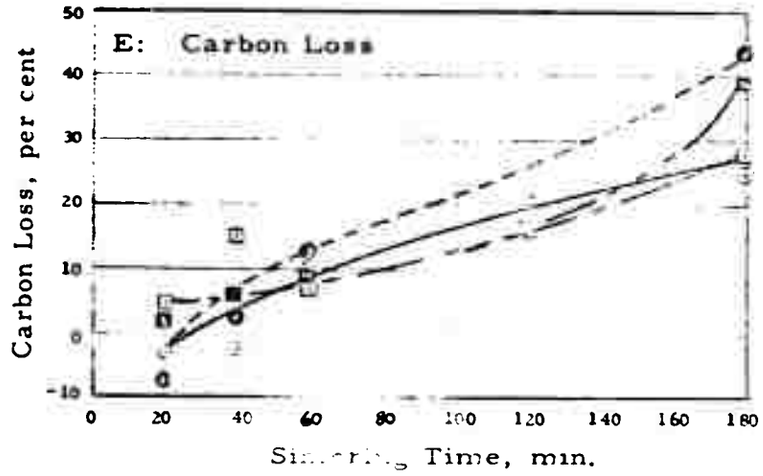


Figure 12D Effect of Sintering Time and Temperature on B, C and H Losses of Sugar-Water Bound Tablets (Continued)

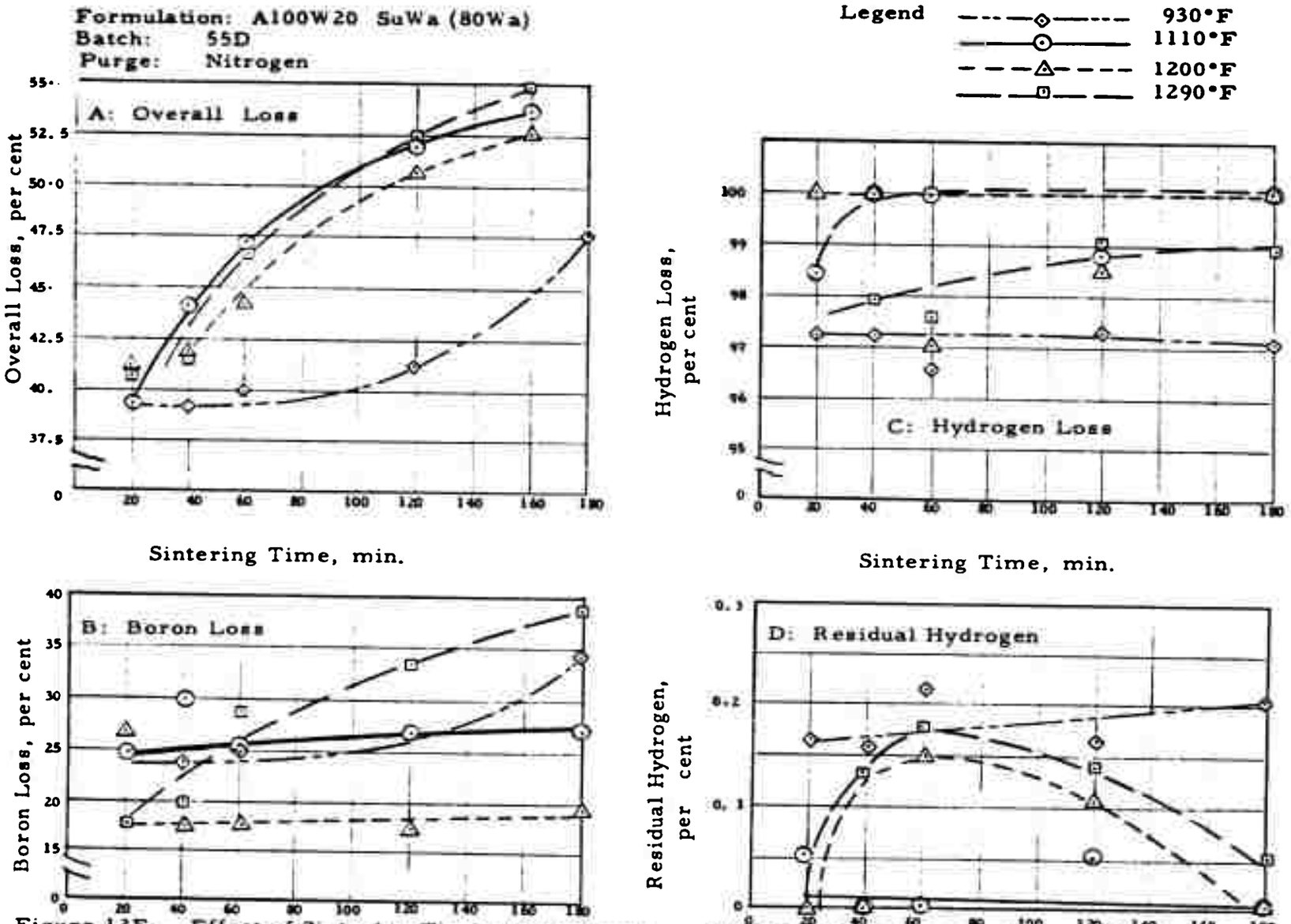


Figure 12E. Effect of Sintering Time and Temperature on B, C, and H Losses of Sugar-Water Bound Tablets.

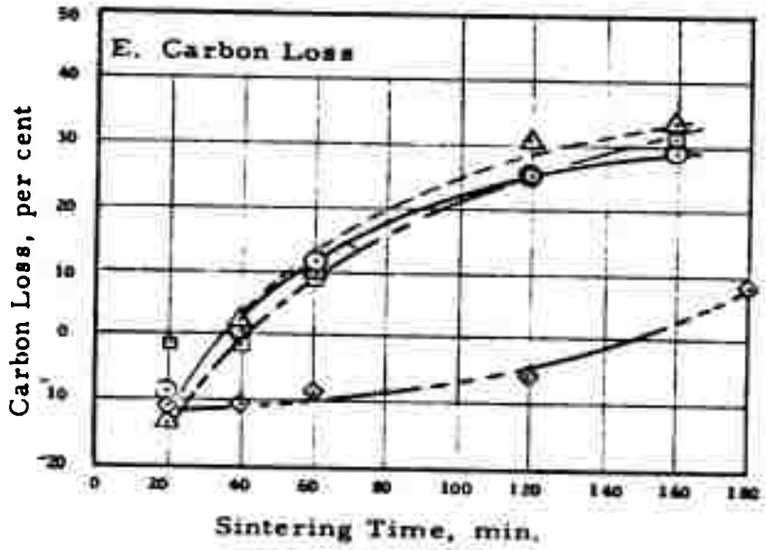


Figure 12E Effect of Sintering Time and Temperature on B, C, and H Losses of Sugar-Water Bound Tablets. (Continued)

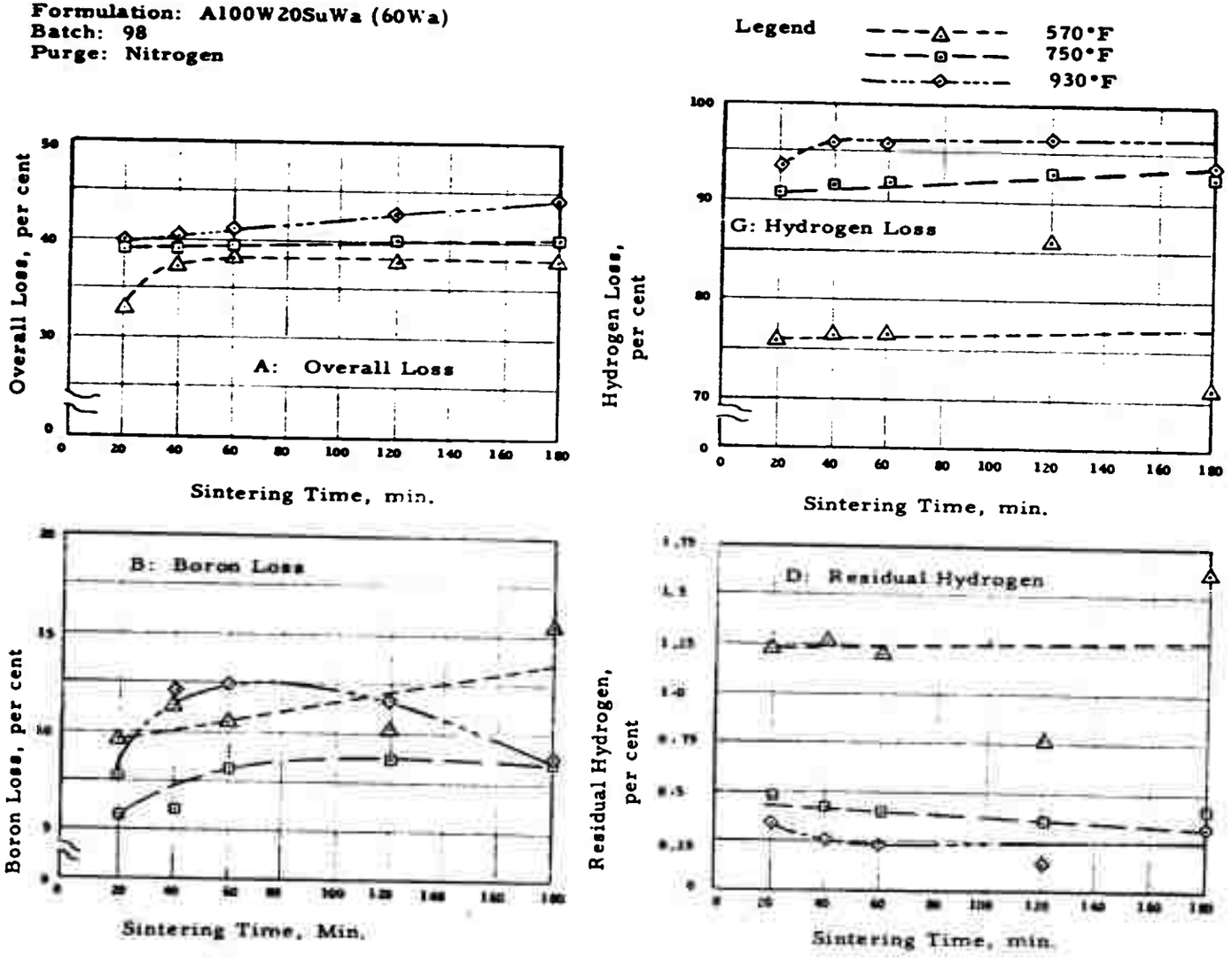


Figure 12F Effect of Sintering Time and Temperature on B, C and H Losses of Sugar-Water Bound Tablets

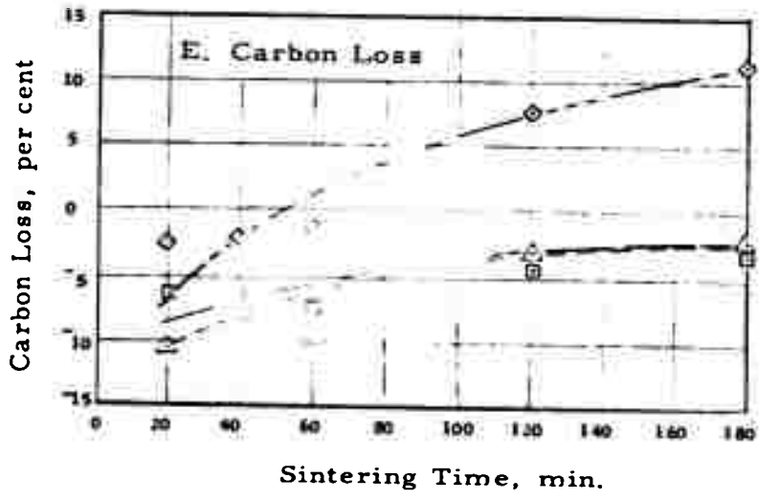


Figure 12F Effect of Sintering Time and Temperature on B, C and H Losses of Sugar-Water Bound Tablets (Continued)

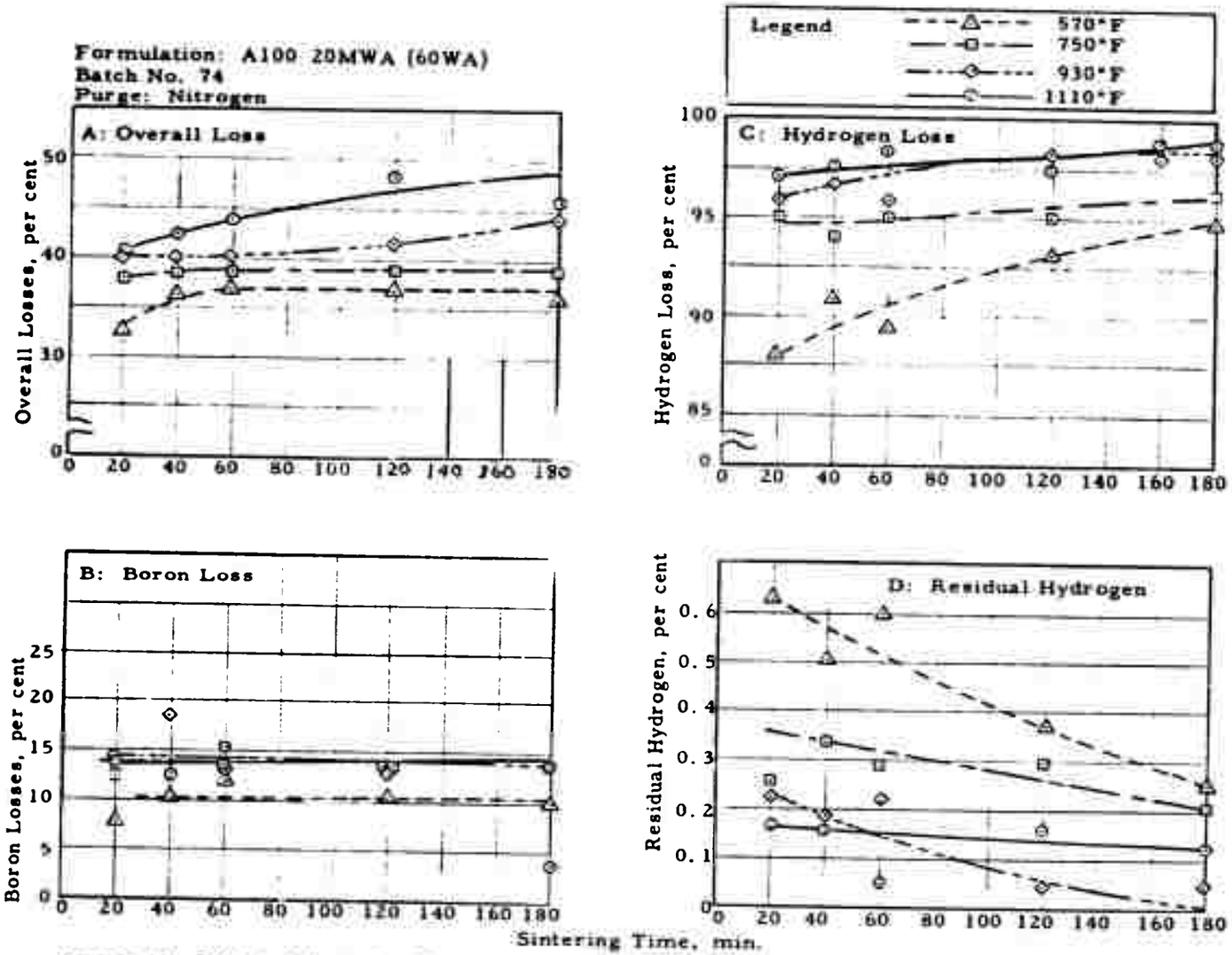


Figure 13 - Effectof Sintering Time and Temperature on B, C. and H Losses of Molasses-Water Bound Tablets

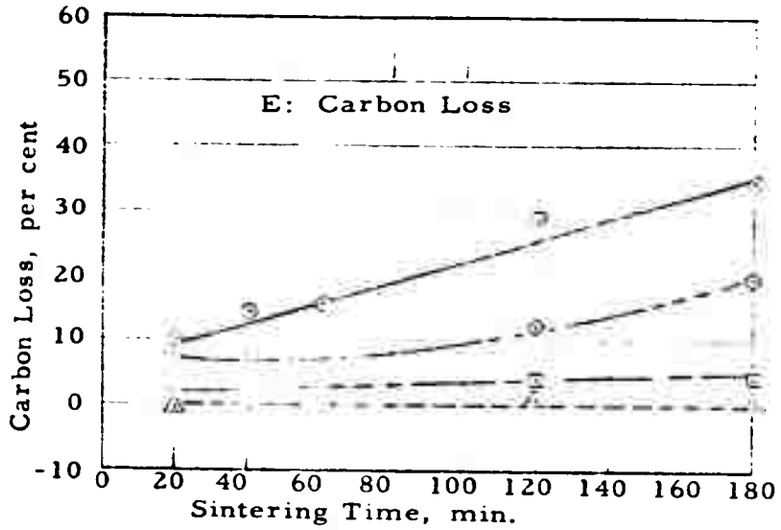


Figure 13 (Continued) - Effect of Sintering Time and Temperature on B, C, and H Losses of Molasses-Water Bound Tablets

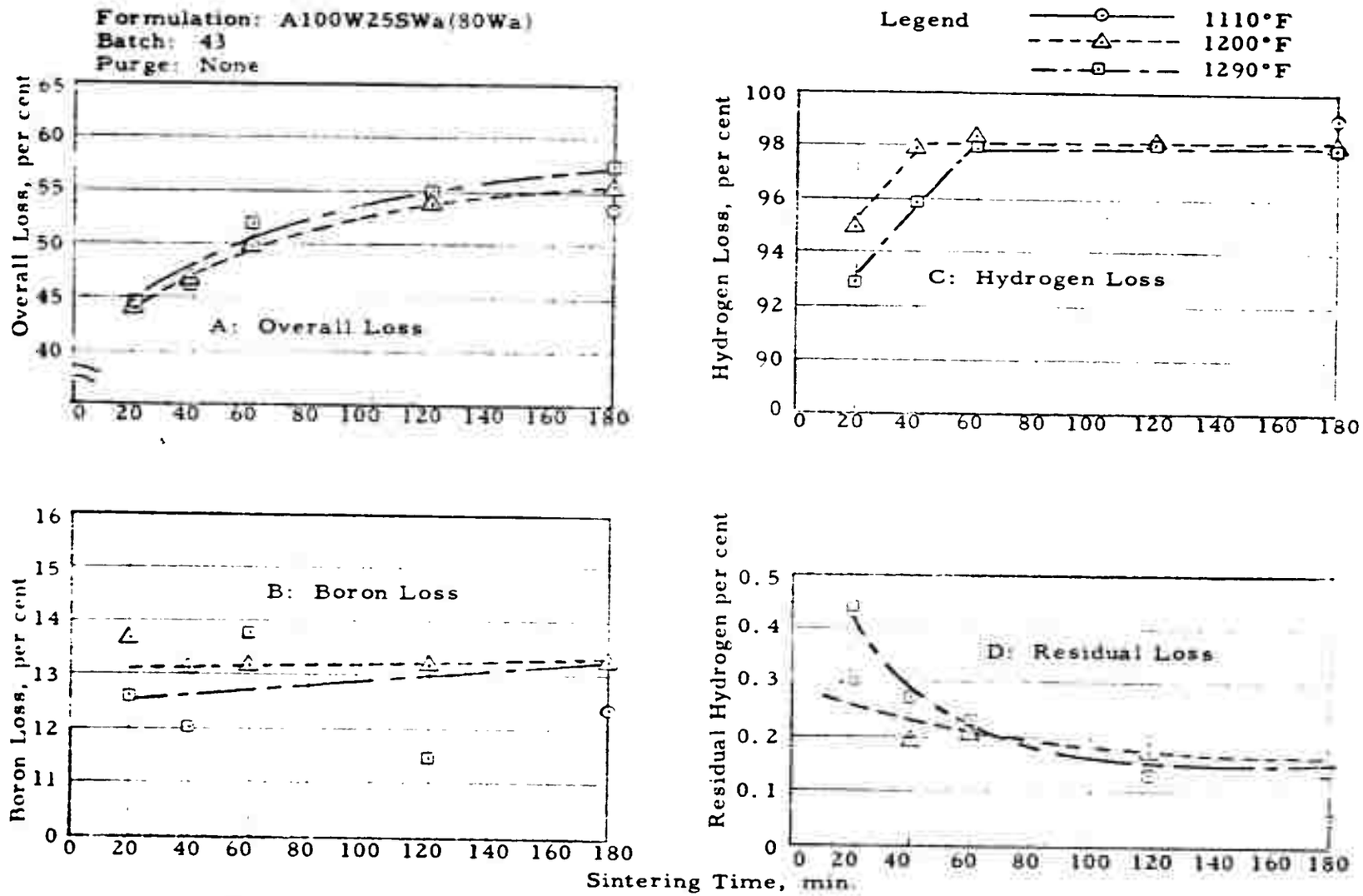


Figure 14A - Effect of Sintering Time and Temperature on B, C, and H Losses of Starch-Water Bound Tablets

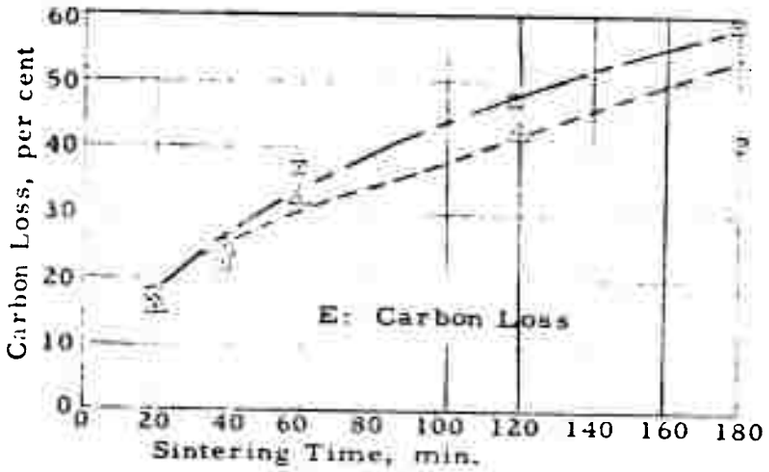


Figure 14A (Continued) - Effect of Sintering Time and Temperature on B, C, and H Losses of Starch-Water Bound Tablets

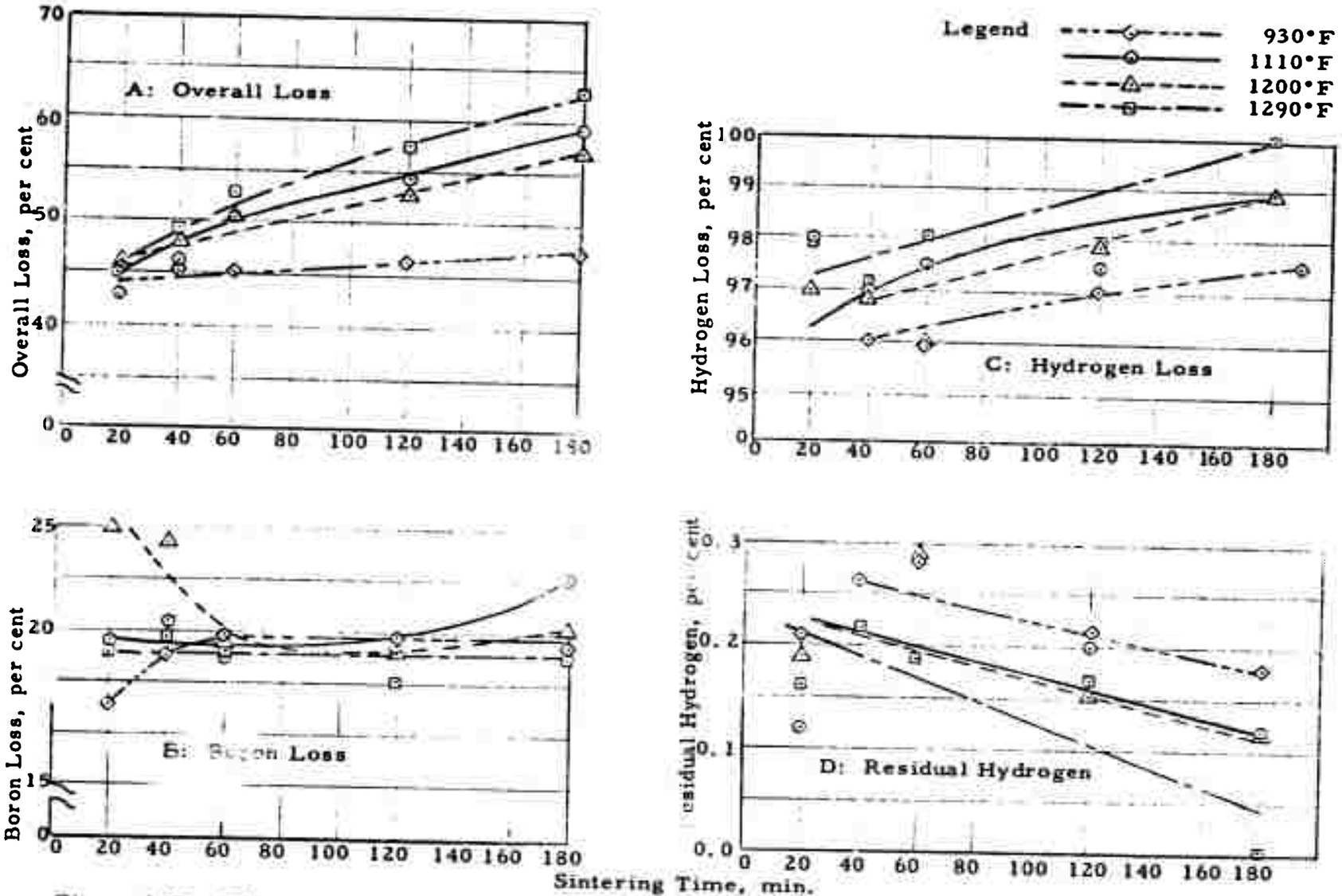


Figure 14B - Effect of Sintering Time and Temperature on B, C, and H Losses of Starch-Water Bound Tablets

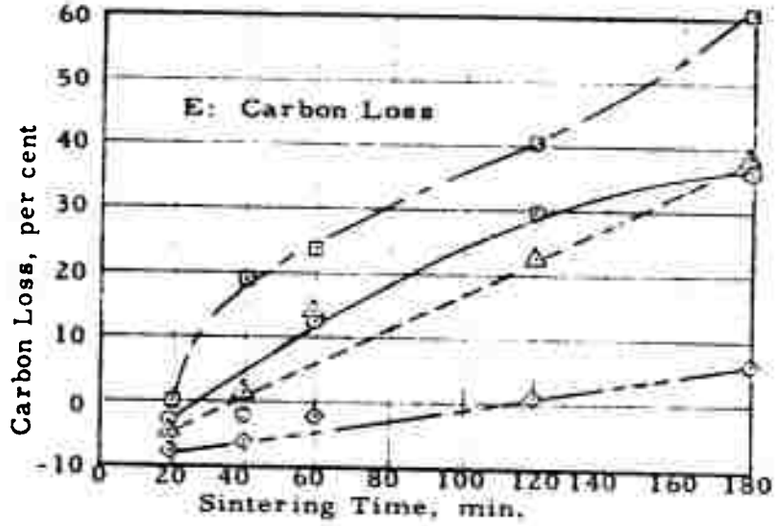


Figure 14B (Continued) - Effect of Sintering Time and Temperature on B, C, and H Losses of Starch-Water Bound Tablets

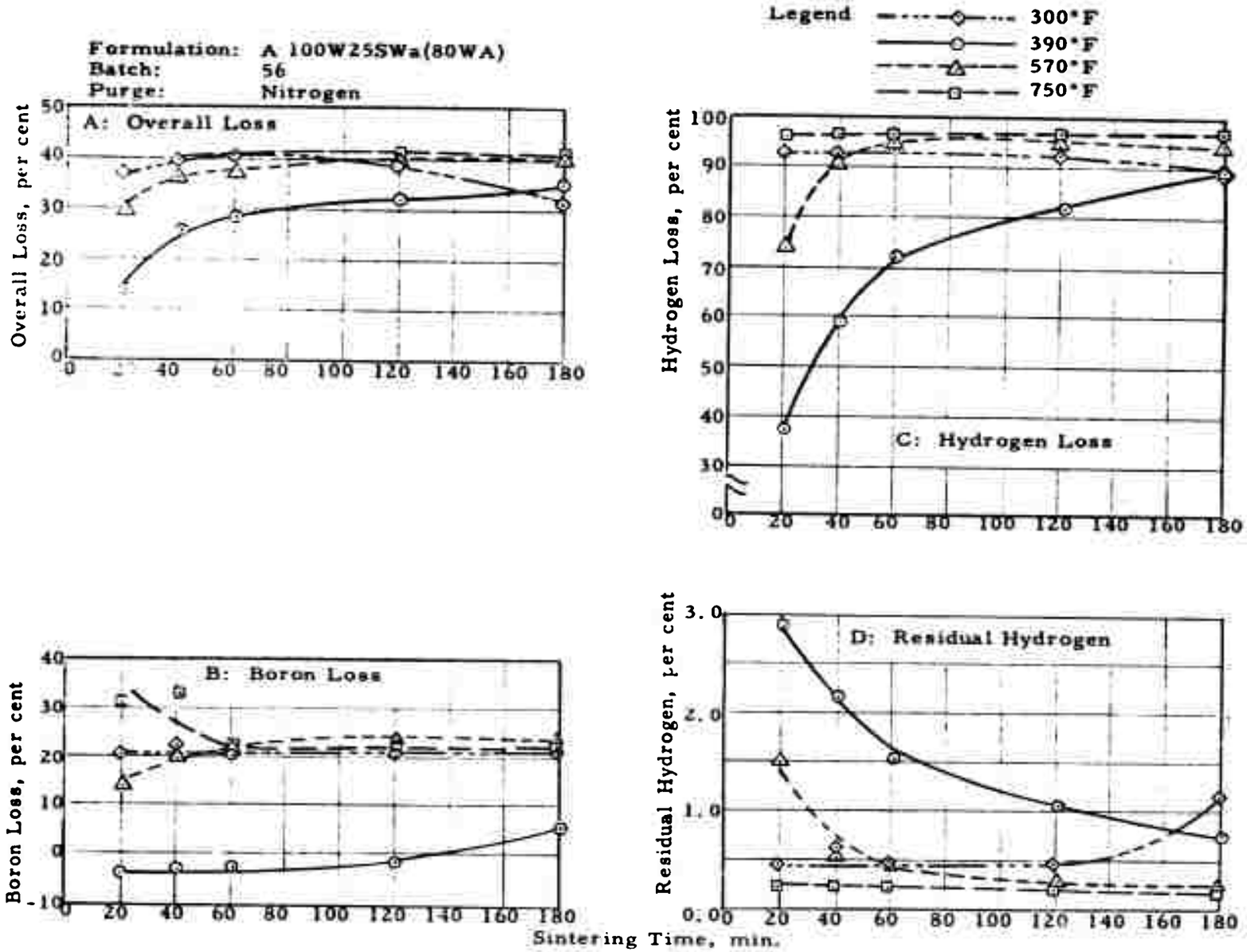


Figure 14C - Effect of Sintering Time and Temperature on B, C, and H.Losses of Starch-Water Bound Tablets.

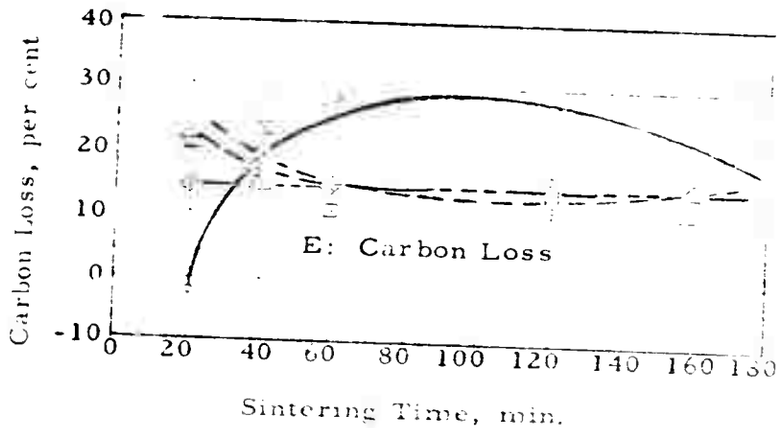


Figure 14C (Continued) - Effect of Sintering Time and Temperature on B, C, and H. Losses of Starch-Water Bound Tablets.

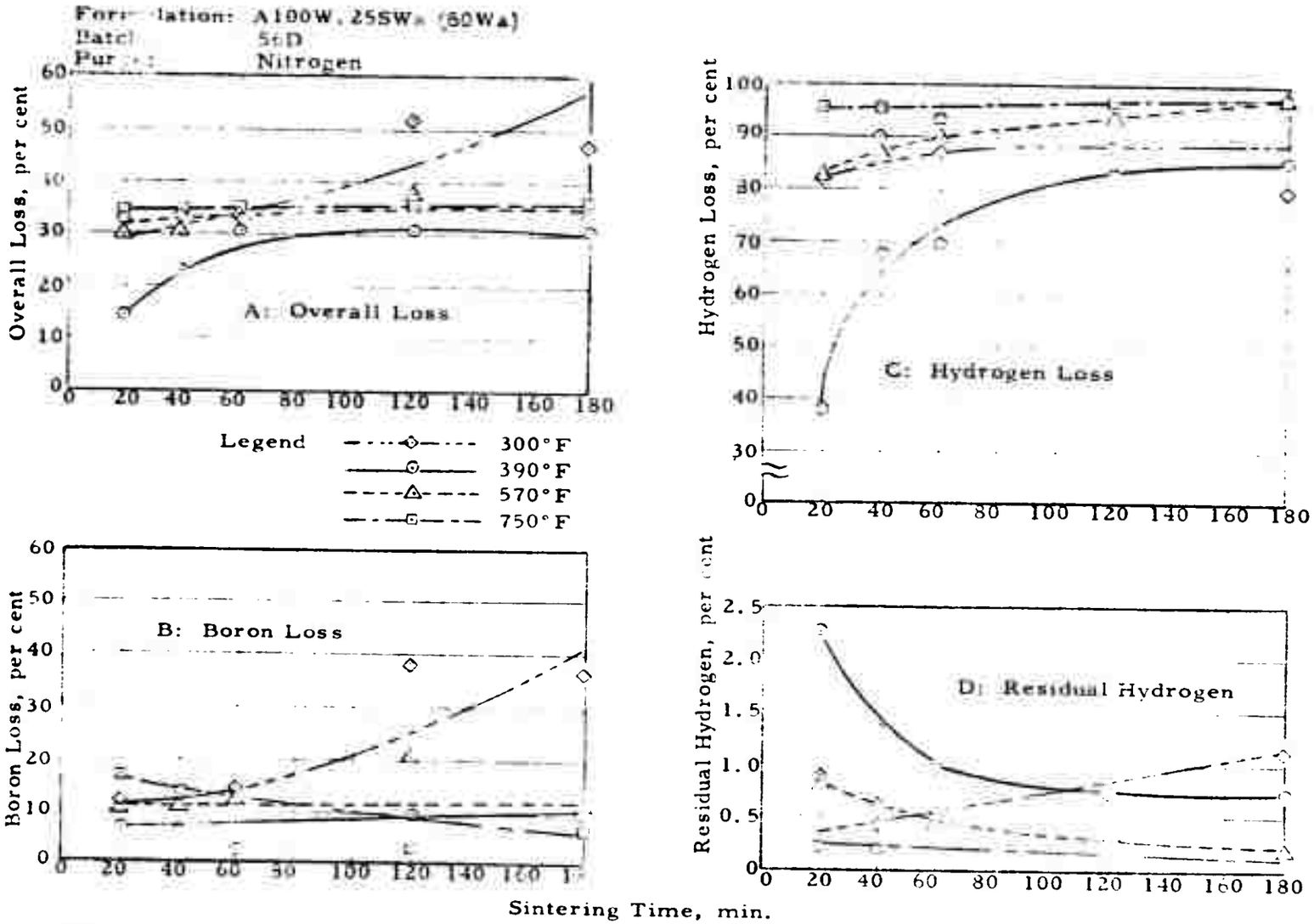


Figure 14D - Effect of Sintering Time and Temperature on B, C, and H Losses of Starch-Water Bound Tablets

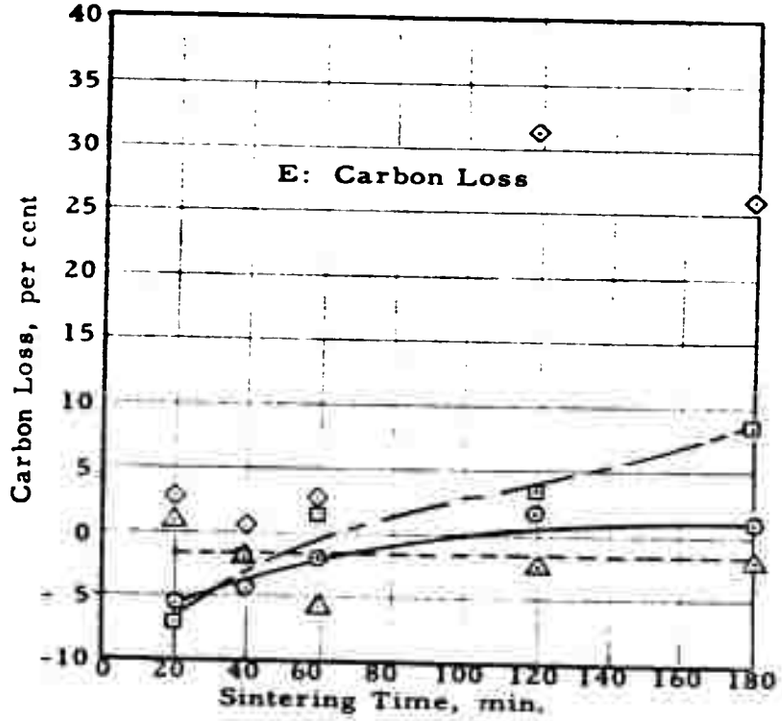


Figure 14D (Continued) - Effect of Sintering Time and Temperature on B, C, and H Losses of Starch-Water Bound Tablets

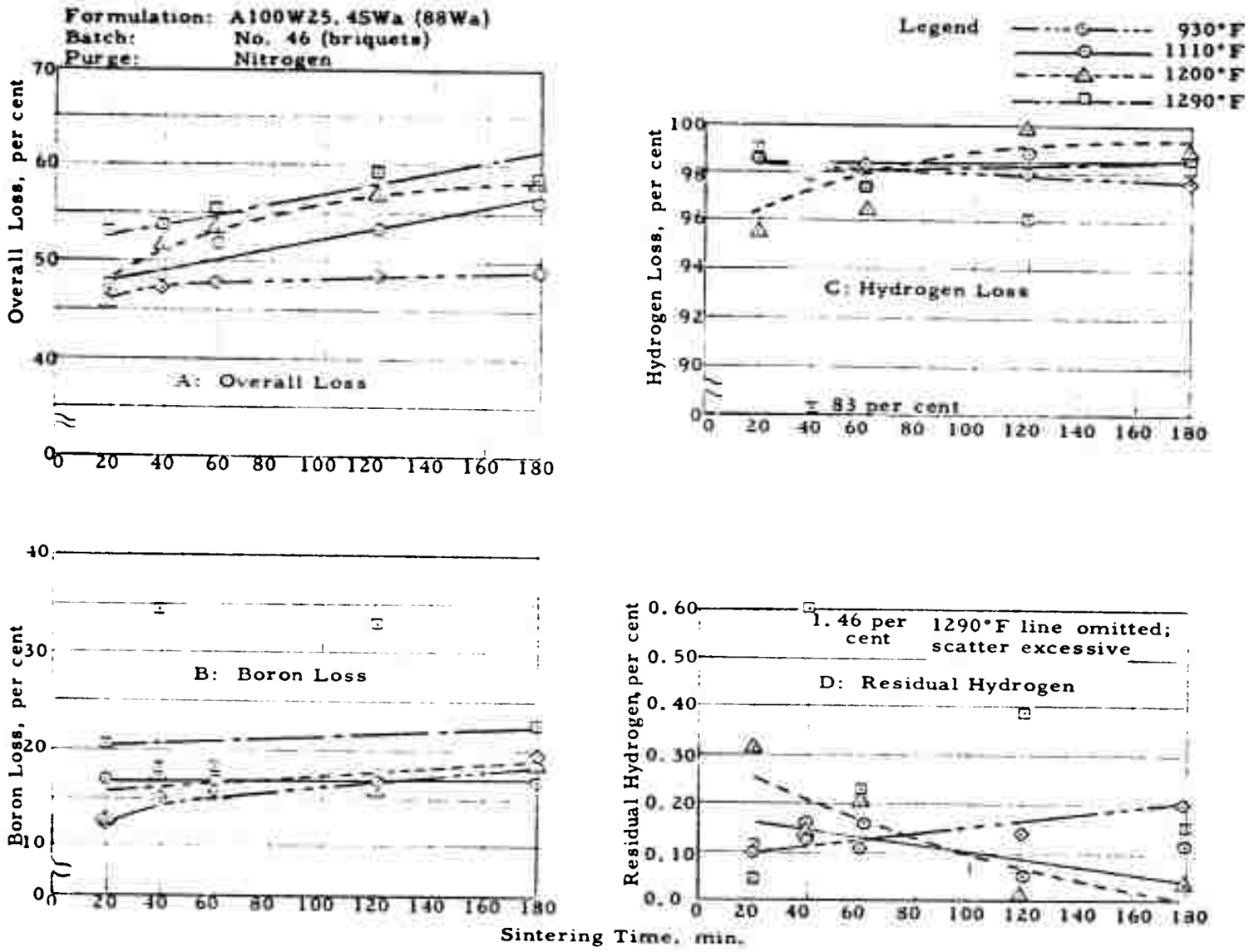


Figure 15 - Effect of Sintering Time and Temperature on B, C, and H Losses of Starch-Water Bound Briquets

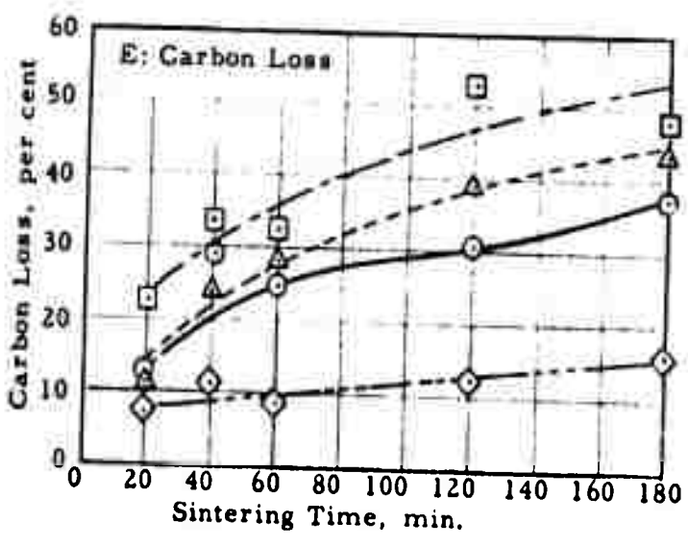


Figure 15 (Continued) - Effect of Sintering Time and Temperature on B, C, and H Losses of Starch-Water Bound Briquets

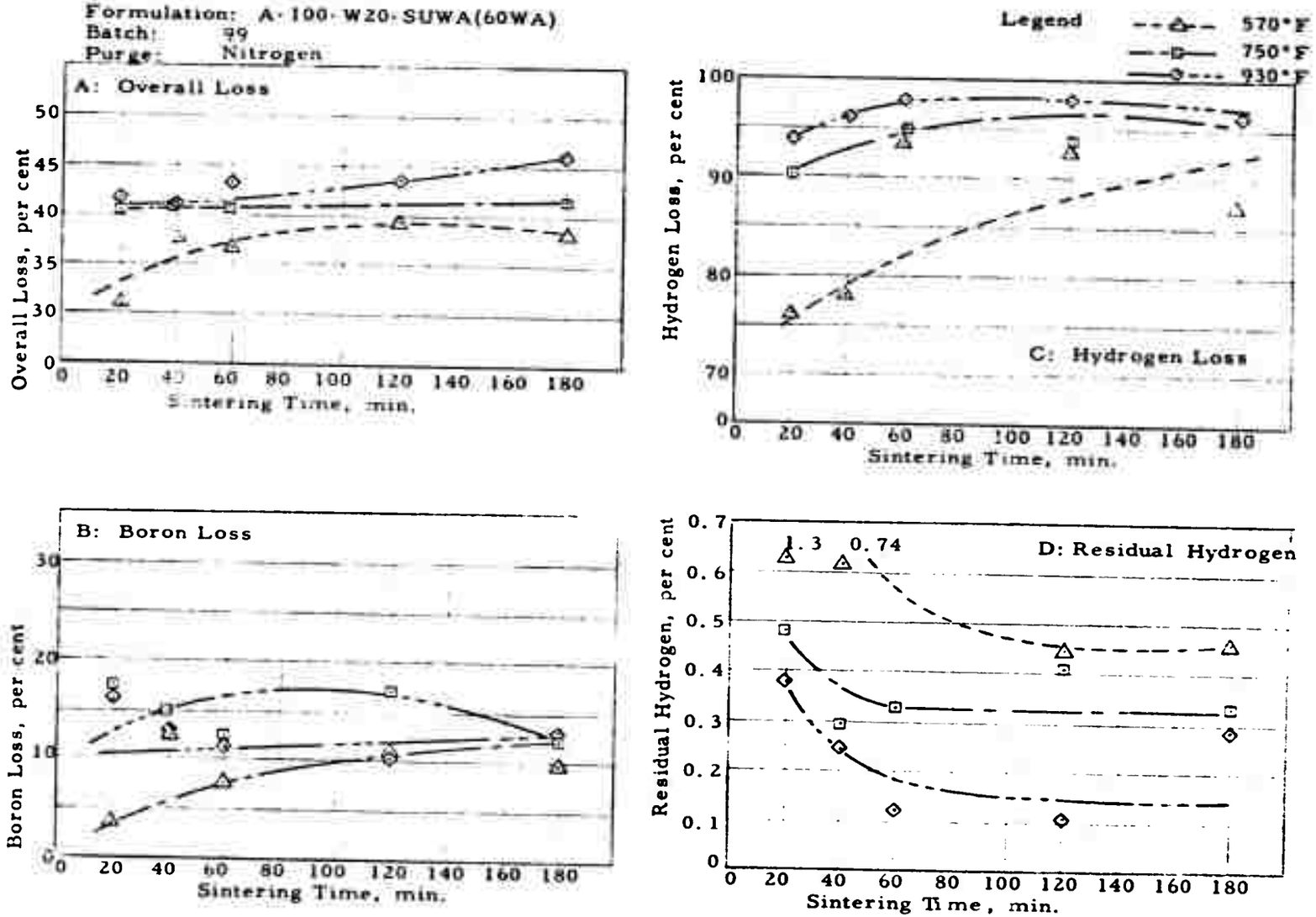


Figure 16 - Effect on Sintering Time and Temperature on Boron, Carbon, Hydrogen and Overall Losses of Sugar-Water Bound Briquets

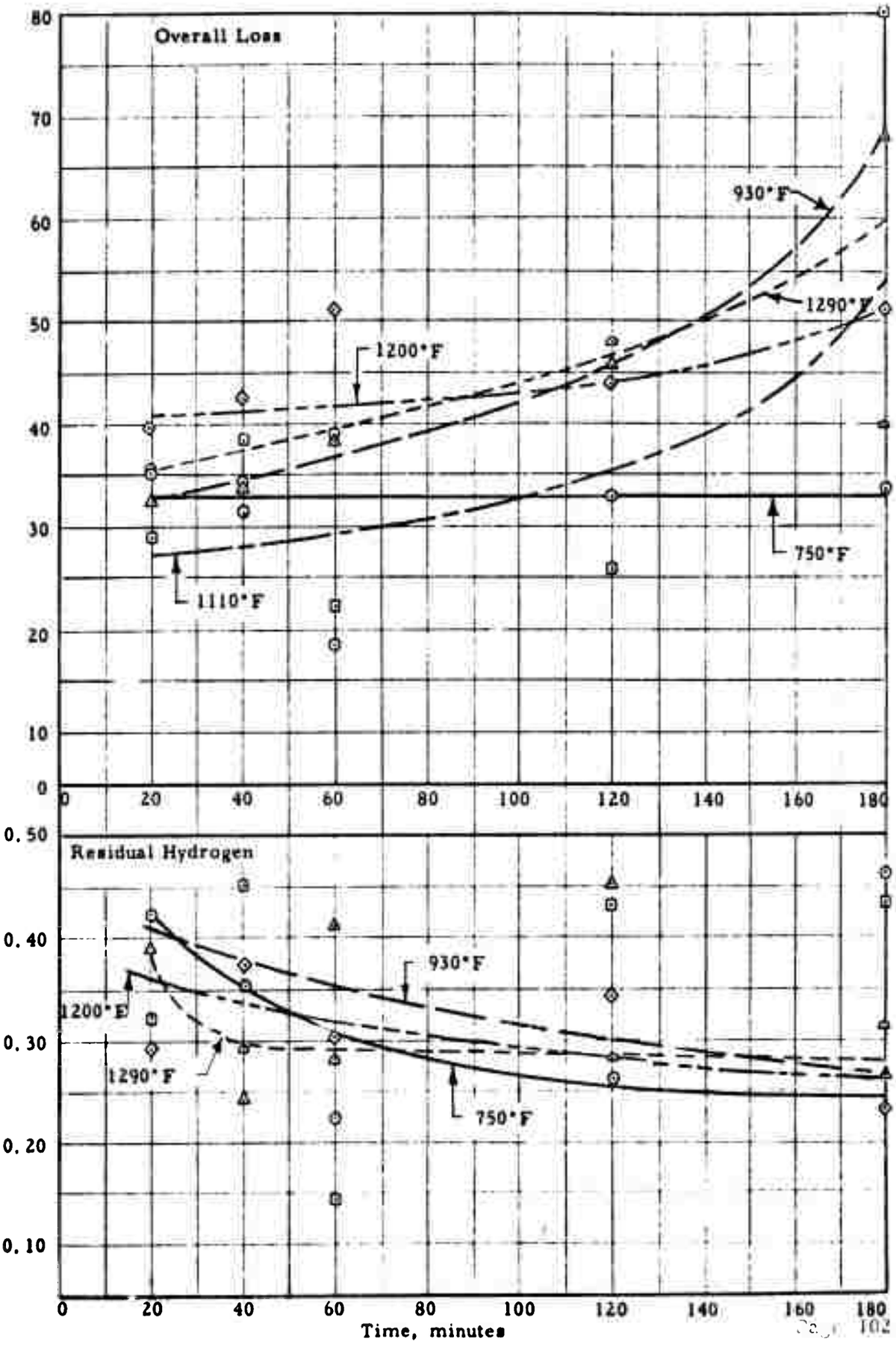


Figure 17 - Effect of Sintering Time and Temperature on Overall Losses of Sugar-Water Bound Carbon Tablets

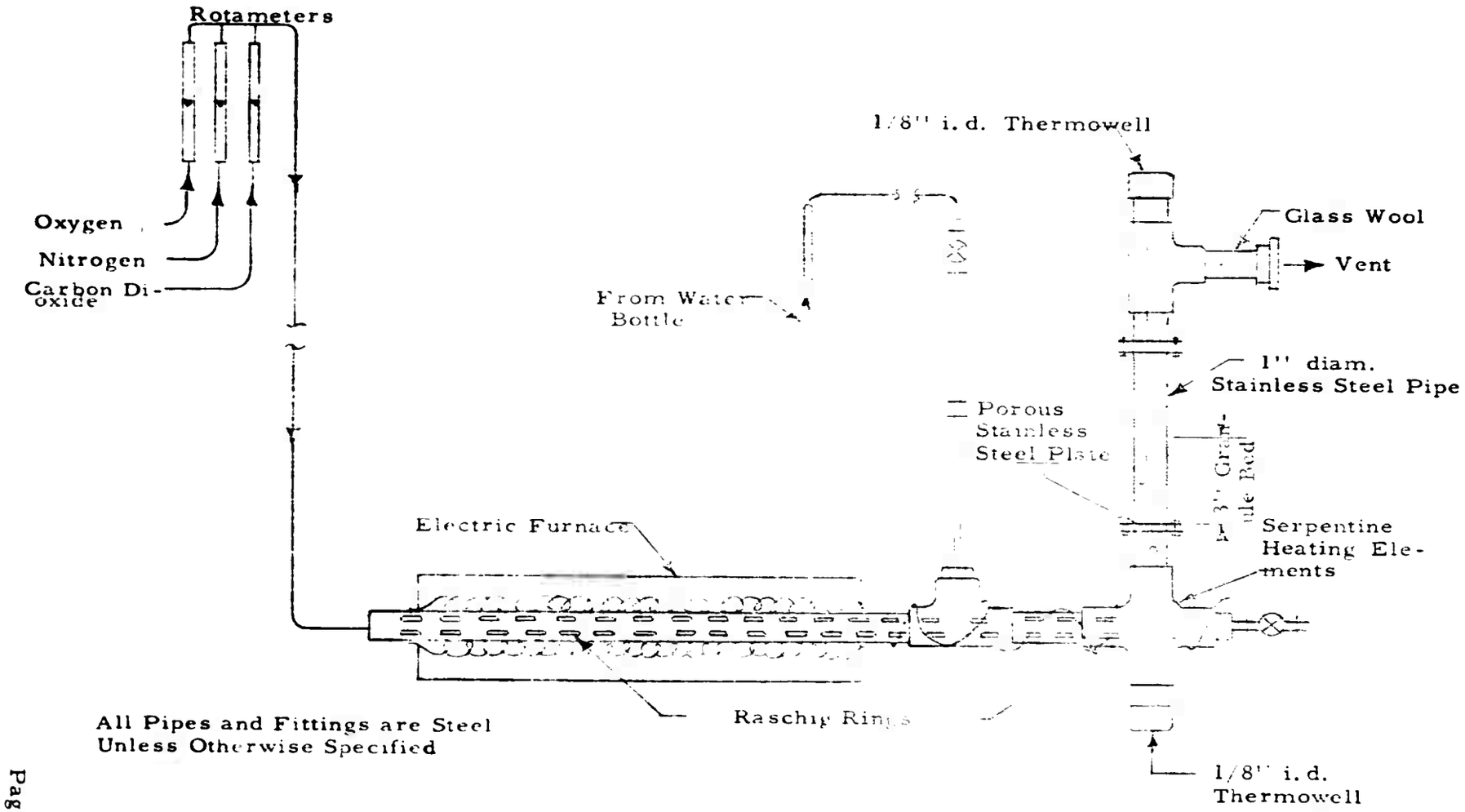


Figure 18 - Apparatus for Feed Material Oxidation Test

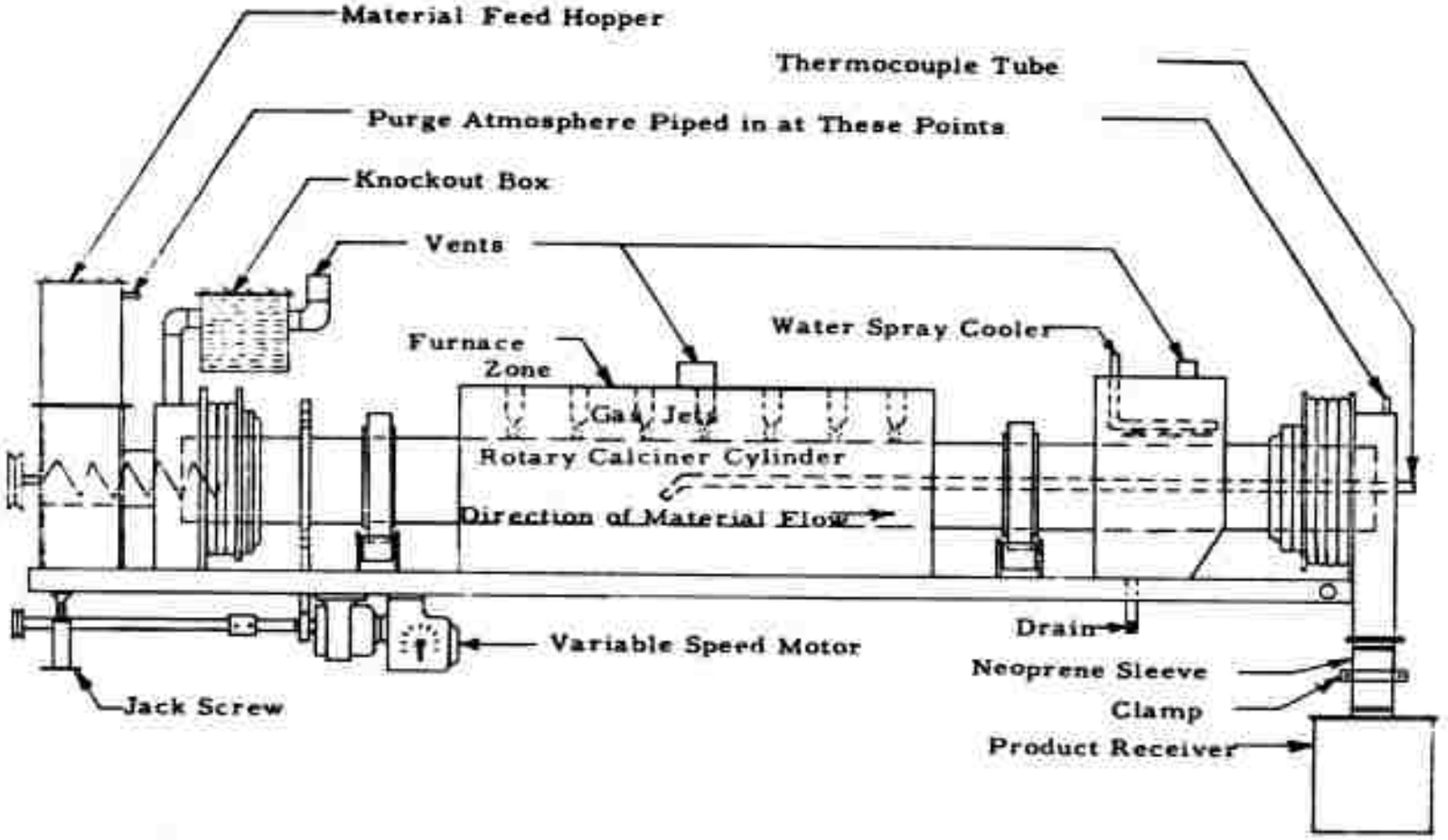


Figure 19 - Rotary Calciner

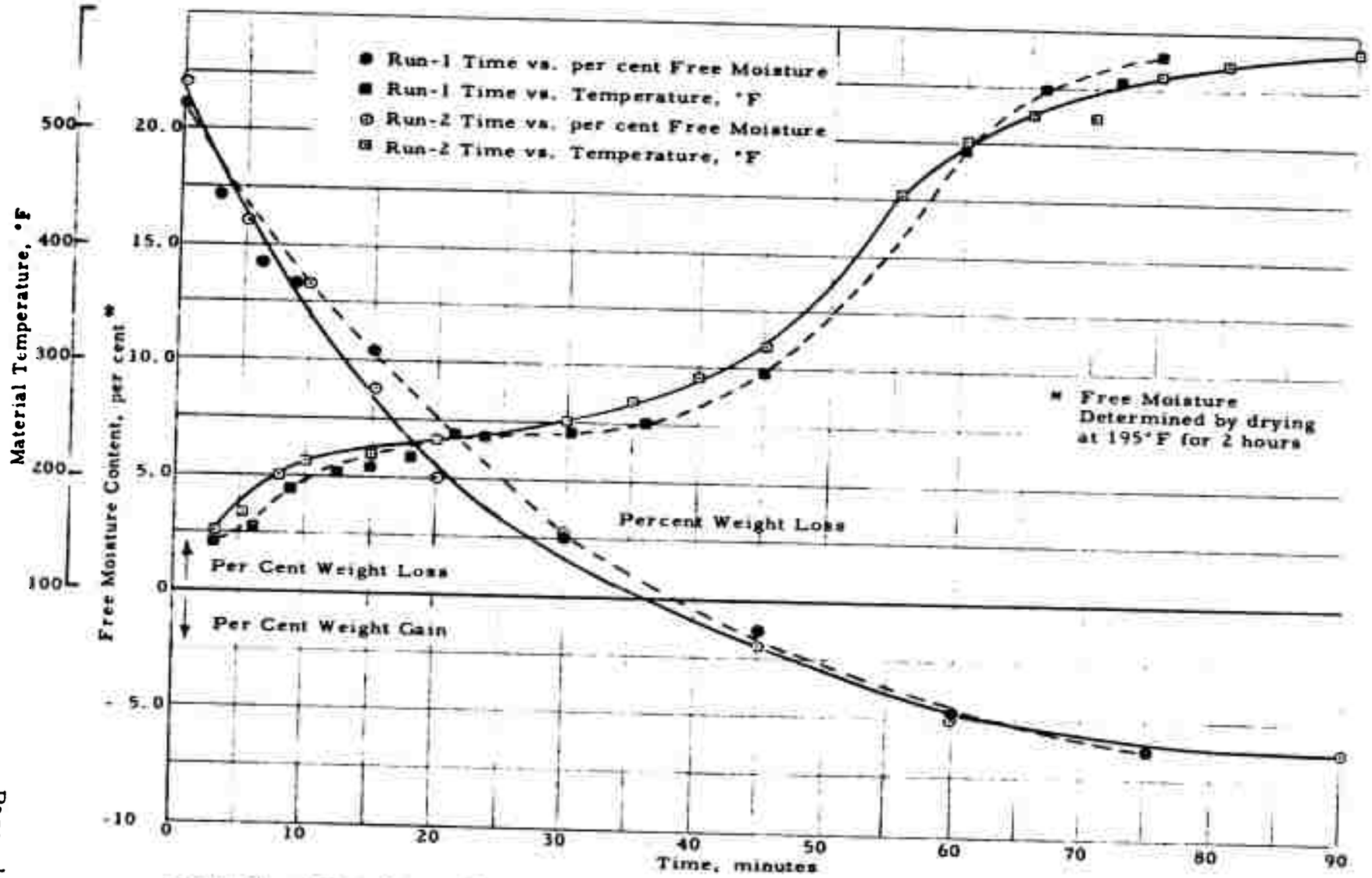


Figure 20 - Drying Curves for Boric Acid-Witco Carbon Granules, Link Belt Co. Tests

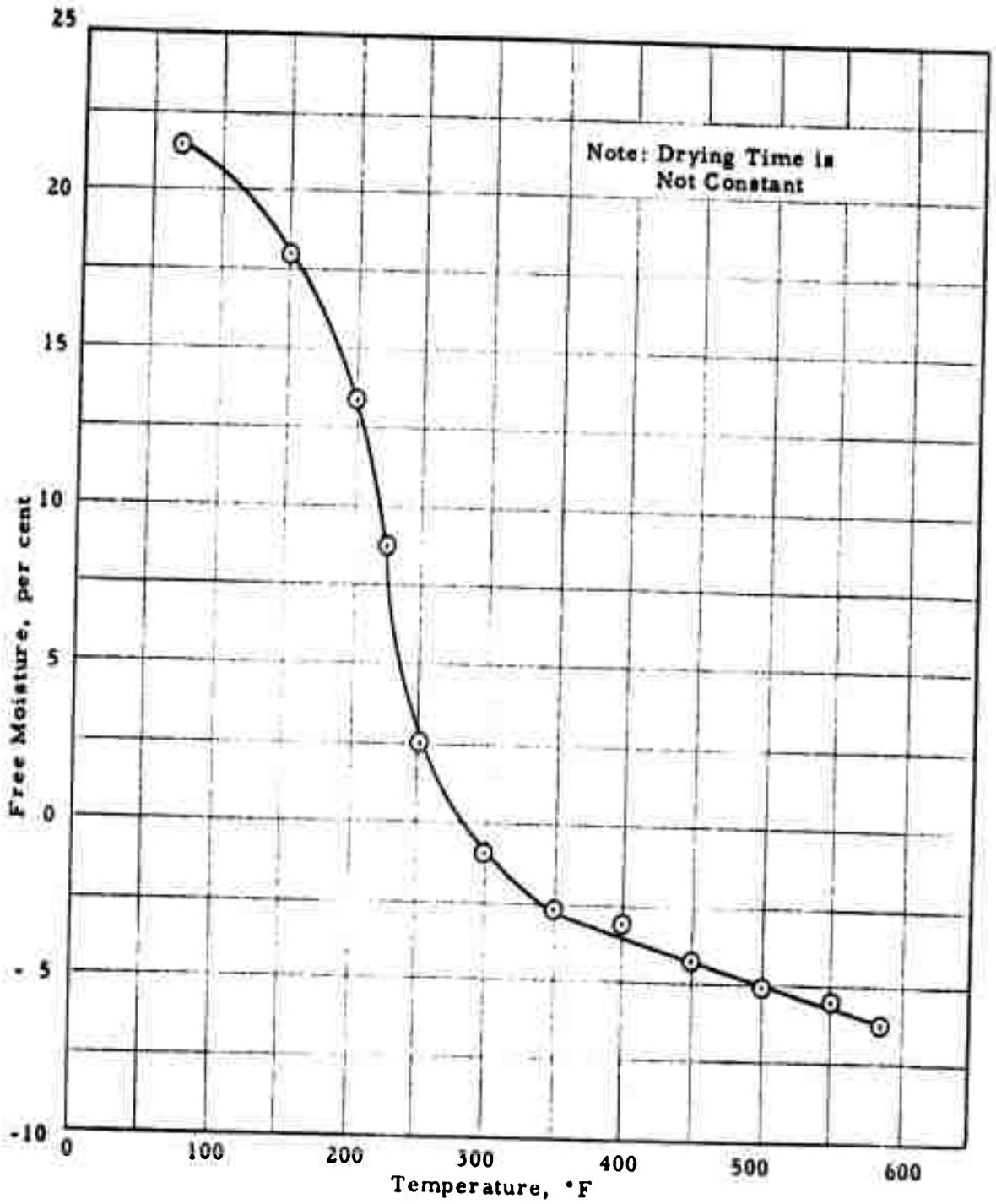


Figure 21 - Free Moisture Versus Drying Temperature for Boric Acid-Witco Carbon Granules, Link Belt Co. Tests

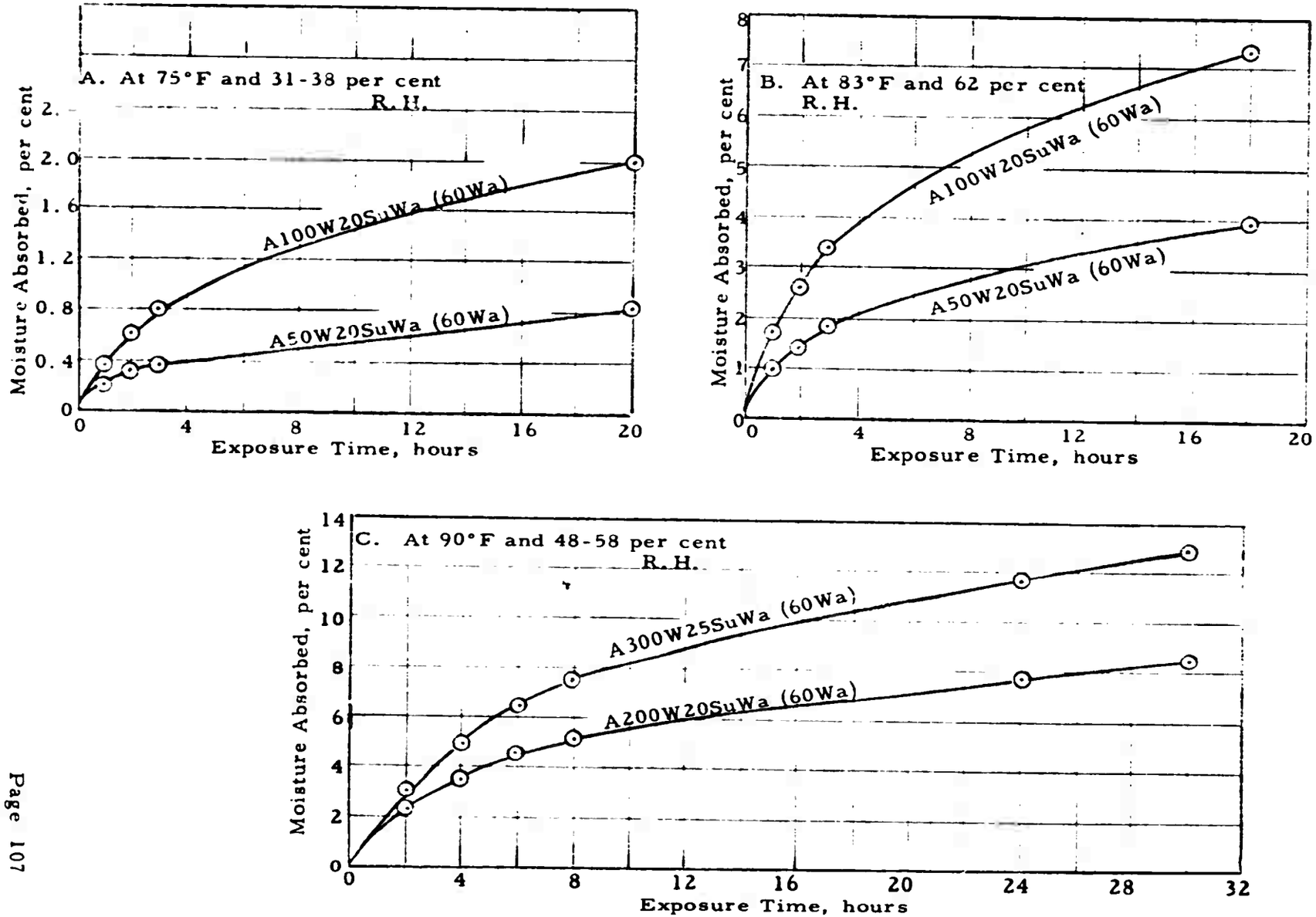


Figure 22 - Moisture Absorption Rate of Boric Oxide-Carbon Briquets

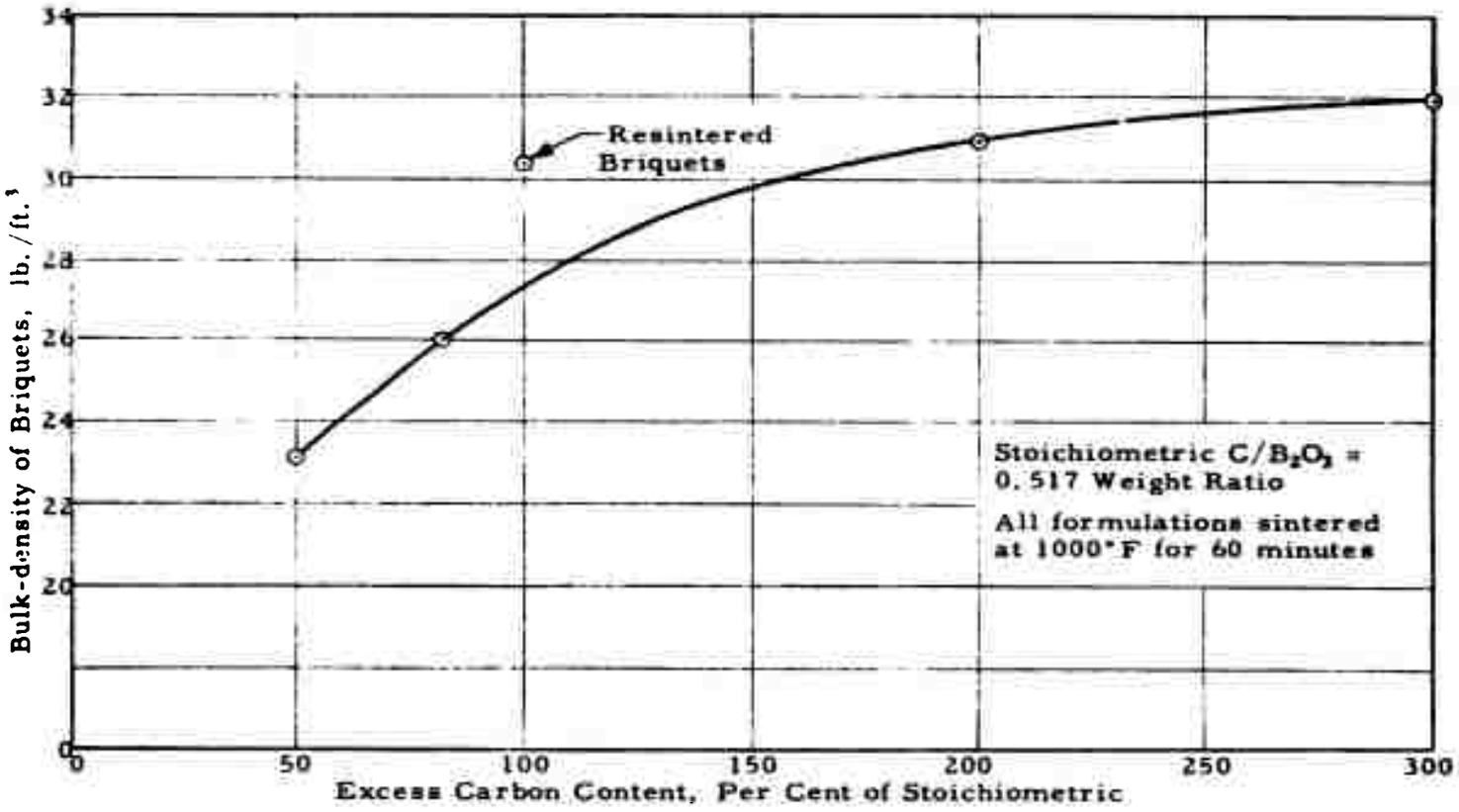


Figure 23 - Effect of Formulation on Bulk-Density of Sintered Boric Oxide-Carbon Briquets

VII. APPENDIX

A. List of Raw Materials

1. Carbon

a. Witco Furnace Black:

(1) Vendor:

Witco Chemical Co.
260 Madison Avenue
New York 16, New York

(2) Chemical and Physical Data

(a) Prepilot Scale Stock

F-1 Witco Powder

Fixed Carbon	99 Per cent
Moisture, maximum	4 Per cent *
Avg. Particle Size, millimicroms	70 mμ
Specific Gravity	1.90
Apparent density	20 lb./ft.^{3}
Ash	0.2 Per cent

(b) Pilot Plant Stock

SRF beads: This form of Witco black is identical with F-1 powder except that it is in beaded (approximately 1/6 inch diameter) form

Fixed Carbon	99 Per cent
Moisture	4 Per cent*
Avg. particle size (ultimate)	70 mμ
Specific Gravity	1.80
Apparent Density	27-30 lb./ft.^{3}
Ash at 550°C	0.35 Per cent

b. Calcined Gilsonite

(1) Vendor:

American Gilsonite Co.
134 W. Broadway
Salt Lake City, Utah

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* Typical = 1.0 Per cent

(2) Chemical and Physical Properties

Ash	< 0.5 Per cent
Volatile	0.5 Per cent
Fixed Carbon	97.5 Per cent
Moisture (typical)	1.0 Per cent
Bulk density, approx.	1.14 lb/ft.^{3}
True density	2.04 lb/ft.^{3}

c. Petroleum Coke

(1) Vendor:

Great Lakes Carbon Corp.
Niagara Falls, New York

(2) Chemical and Physical Properties

Type 3005 and 3020:

Fixed carbon	98.0 Per cent +
H_2O	0.1 Per cent
Sulfur	1.0 Per cent
Ash	0.25 Per cent

SCREEN TEST

Type 3005	
mesh size	Per cent Retained
1/2 in.	4
3/8 in.	9
3 m.	10
4	12
6	12
10	25
20	18
35	6
65	1
thru 200	3

Type 3020	
mesh size	Per cent Retained
150	Trace
200	3
Pan	97

2. Boron Compounds:

a. Boric Oxide:

60 and 100 mesh technical powder:

(1) Vendor:

U. S. Borax and Chemical Corp.
50 Rockefeller Plaza
New York 20, New York

(2) Chemical and Physical Properties

B_2O_3		98.83 Per cent
H_2O		0.75
Ash:	maximum	0.77
	typical	0.32
Bulk density		
	60 mesh	56. lb/ft.^{3}
	100 mesh	61.5 lb/ft.^{3}

b. Boric Acid:

60 and 100 mesh technical grade powder:
20 mesh granules:

(1) Vendor:

U. S. Borax and Chemical Corp.

(2) Chemical and Physical Properties

Theoretical Composition

B_2O_3	56.3 Per cent
H_2O	43.7
Impurities	nearly the same as B_2O_3

c. Anhydrous Sodium Tetraborate

(Anhydrous Borax Dust)

(1) Vendor:

U. S. Borax and Chemical Corp.

(2) Chemical and Physical Data

Theoretical Composition:

Na_2O	30.8 Per cent
B_2O_3	69.2 Per cent

Typical Analyses

$Na_2B_4O_7$	99.3
H_2O	0.4
Ash	0.3

3. Binders:

a. K-150

Koppers petroleum pitch

m.p. = 150°F

Koppers Chemical Co.

b. K-200

Koppers petroleum pitch

m.p. = 200°F

c. Refined (white, table) Sugar:

purchased in 5 and 100 lb. cartons or bags from several wholesale and retail grocers

d. Unrefined (brown) Sugar:

purchased in 2 and 5 lb. size cartons from several retail grocers

e. Molasses, black strap, Industrial:

Riverside Chemical Co.
North Tonawanda, New York

Total ash		8.4 Per cent
CaO	1.5	
K_2O	3.5	
H_2SO_4 (SO_3)	1.6	
Na_2O, Fe_2O_3	0.2	

f. Starch, Potato, commercial

Morningstar-Nicol